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MINISTRY OF STATE LABOR RESERVES USSR

P. P. Karpov

PERERABOTKA NEFTI [PETROLEUM REFINING]

Approved by the Academic Council of Technical-Vocational Education of the Ministry of State Labor Reserves USSR as a text-book for petroleum-refinery operators

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Note

This book contains information on the general properties of petroleum products, on the methods of primary distillation, thermal and catalytic cracking, on the methods of chemical processing of hydrocarbon gases, refining of dark petroleum products, and the production of oils.

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INTRODUCTION

PETROLEUM AND NATURAL GAS AS MINERAL FUELS

Mineral fuels include coal, peat, shales, asphalt, petroleum and natural gas.

All of these substances consist chiefly of carbon, hydrogen, oxygen, sulfur and nitrogen, and are organic substances containing from 60 to 95 percent carbon (peat having the lowest carbon content and anthracite the highest), hydrogen, from 2.5 percent for peat to 14 percent for petroleum, and oxygen, nitrogen and sulfur, from fractions of a percent for petroleum to 30-35 percent for peat.

TABLE 1

ULTIMATE ANALYSIS OF MINERAL FUELS (IN PERCENT)

	C	H	S+O+N
Anthracite	95.0	2.5	2.5
Coal	85.0	5.4	9.6
Brown coal	68.0	5.5	26.5
Peat	60.0	6.2	33.8
Light petroleum	85.3	14.2	0.5
Heavy petroleum	86.5	11.0	2.5

Among the other mineral fuels, the most valuable are petroleum and natural gas, which are used as fuel and as raw material for processing into various products.

Petroleum has been known since antiquity. In excavations of the most ancient cities in Egypt, lamps have been found with solidified petroleum residues. The ancient Greeks used petroleum as an

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illuminant, a lubricant, and for medicinal purposes. They also employed it for military purposes, for throwing the so-called Greek fire, or vessels filled with burning petroleum.

For many centuries petroleum was only used by the inhabitants of petroliferous regions, who obtained it from wells and used it in the unrefined form as an illuminant and for medical purposes.

In 1745 Nabatov built the first petroleum refinery in the world on the Ukhta. In 1823 the Dubinin brothers, who were feudal peasants, built a plant for the processing of petroleum to kerosene at the city of Mozdok, North Caucasus (Figure 1).

In 1854 the first industrial oil well was drilled in Russia on the Ukhta.

In 1901 Russia advanced to the first place in world oil production with a total yield of 11.5 million tons (51 percent of world production), but in 1913 its production dropped to 9.2 million tons. The capitalist oil well owners rapaciously exploited deposits and the technology of the oil industry was extremely backward.

Only after the nationalization of the oil industry did rapid development commence: by 1927 petroleum production had reached its 1913 level again, while in 1940 this level was exceeded three times.

With the appearance of new types of engines and machines, the prevailing ideas on the value of petroleum products were correspondingly modified. Thus kerosene, which was in demand as an illuminant, was the principal product of petroleum refining during the early days of the expansion of the oil industry. Fuel oil and gasoline were of no industrial use at that time and were regarded as troublesome waste

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material involving the hazard of fire; they were taken outside the city and disposed of by burning in pits.

In the 70's, with the invention of the jet burner, fuel oil was recognized as a valuable and convenient form of fuel for firing steam boilers. After the invention of the internal combustion engine, and the appearance of automobiles and airplanes, gasoline became the product of primary importance. The thought of oilmen began to be directed towards the development of new methods of producing gasolines in larger quantities, and of better quality. During the last ten or fifteen years chemical methods of manufacturing high-grade aviation gasolines have been very rapidly introduced into the oil-refining industry.

But tractor fuel, lubricating oil, Diesel fuel, boiler fuel, etc, also acquired immense importance.

At the present time the oil refining industry produces over 100 varieties of petroleum products used in the various branches of the national economy and military technology.

THE PRESENT SIGNIFICANCE OF PETROLEUM IN WORLD ECONOMY

Thanks to its high heat value, its convenient transportation, and to the simplicity of its combustion, which requires only small numbers of service personnel, petroleum is an exceptionally high-grade fuel. The heat value of petroleum fuel reaches 10,000 calorie-kilograms and over. This means that 10,000 calories of heat are released with the burning of 1 kilogram of fuel. Other forms of fuel have considerably lower heat values; to obtain the same amount of heat, 1½ times as much anthracite is needed, twice as much peat and 2-2½ times as much wood or straw (Figure 3).

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This explains the fact that petroleum fuel is gradually displacing other forms of fuel in industry, especially in transportation. In 1914 the oil-burning motor-ships and steamships of Russia comprised only 4 percent of the entire merchant marine, but by 1939 this percentage had risen to 58. The navy operates exclusively on oil fuel. This is only natural, since coal firing would require a large number of stokers, besides a number of other inconveniences, i.e., dirt on the ship, the need for carrying large supplies of coal. Thus, a ship of the line might travel for 15 days on a full load of coal, while the same ship of the line, if it burned oil instead, could take on a supply sufficient for 57 days of voyaging.

Gaseous fuel is of even higher quality than petroleum fuel: the delivery of gas is accomplished through pipes from the supply point, and the only work connected with the use of a gas burner is opening a cock and lighting the gas.

However, in spite of the high quality of crude oil, it is uneconomic to use it for fuel. With every passing year, crude petroleum is being utilized more rationally and more purposefully.

With the steady growth of the truck fleet, the demand for automobile gasoline increases. Our agriculture is completely mechanized, and hundreds of thousands of tractors and various agricultural machines work in our fields. Civil aviation is becoming more and more important. Everywhere lubricating oils play an immense part: there is not a single moving mechanism, tool nor machine that can operate without lubrication. Even for lubricating

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carts, wheel lubricants prepared from petroleum are needed.

The importance of petroleum products in wartime is even greater: all modern infantry units are moved by truck, artillery by mechanized chassis, while everyone knows the role of tanks and airplanes. It may be said that the motor is the heart of the modern military machine and that petroleum products are its blood.

THE MOST IMPORTANT OIL REGIONS AND OIL DEPOSITS

Petroleum is widely distributed in nature and has been discovered in almost all countries, but the number of large-scale deposits that are commercially exploited is limited.

Among the old deposits in this country that have long been worked, the following should be noted: (a) Baku region; (b) North Caucasus -- Grozniy and Maykop regions; and Dagestan; (c) Emba region, occupying an immense area of Kazakh SSR from the Caspian Sea to the spurs of the Urals; (d) Far East, on Sakhalin Island; (e) Central Asia, in Uzbek and Turkmen SSR; (f) Western Ukraine.

In the Eastern part of the USSR the first petroleum was produced only in 1929, in the Verkhne-Chusovskiye Gorodki; the Ishimbay deposit was discovered afterwards.

In recent years especially great significance has been acquired by the deposits of the Second Baku, located in the regions of Bashkir ASSR (Ishimbay, Tuymazy, Karly, Termen'-Yelga), of Molotov Oblast (Krasnokamsk, Severokamsk, Verkhne-Chusovskiye Gorodki), of Chkalov Oblast (Buguruslan), of Kuybyshev Oblast (Syzran', Stavropol'), and of Tatar ASSR.

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Every year more new deposits are discovered.

CONDITIONS OF DEPOSITION OF PETROLEUM IN THE EARTH'S CRUST

Petroleum is deposited in the so-called sedimentary rocks. Owing to the porosity of the rocks, immense amounts of oil may be accumulated in them. For the most part the oil saturates sandstones (Baku oil) and limestones (Ishimbay).

Petroleum deposits are ordinarily represented by a productive zone consisting of several strata.

Water and gas are the chief substances that accompany oil. Owing to the differences in specific gravities, the water is located at the bottom, the oil in the middle and the gas on the top (Figure 4). For this reason, gas, water and oil may all be encountered on drilling a well.

THE ORIGIN OF PETROLEUM

Coal is a solid. In its diggings we find traces of the substances from which it originated: leaves, branches, twigs and bark of trees. These signs have positively established the origin of coal from the petrified remains of trees.

Petroleum, however, is a liquid, and no traces of its formation can be found in it. There are therefore a number of different hypotheses on its origin. The Russian scientist Mendeleyev first proposed the theory of the inorganic origin of petroleum. He supposed the earth's core to contain molten carbides of various metals, i.e., compounds of metals and carbon. Water seeping into the earth's core through fissures reacts with these carbides, forming

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organic substances, which are displaced from their place of origin towards the earth's surface, are gradually altered, and are finally converted into petroleum.

An organic hypothesis was then proposed, according to which petroleum was formed from animal remains (fishes, lower organisms) buried as a result of elemental natural phenomena. These remains were gradually converted into petroleum under the action of temperature and pressure.

Lastly there is also a hypothesis of the formation of petroleum from plankton -- the simplest aqueous plants (algae) and animals (minute molluscs) or rotting silt. Since both vegetable substances and lower animal organisms participate in the formation of plankton, this hypothesis is termed that of the mixed plant-animal origin of petroleum.

METHODS OF EXTRACTING PETROLEUM

Drilling

To bring petroleum to the surface, a well must be drilled.

The principal method of drilling at the present time is the rotary method. For this purpose a metal derrick is erected in the shape of a truncated pyramid, sometimes as high as 41 meters. At the center of the derrick a revolving platform is set up, which is a plate with a square opening. The leading working pipe, also of square section, is set in the opening. The drilling tool or bit is screwed on the end of the pipe. When the bit turns it cuts through the rock. To remove this rock waste, a clay solution is

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forced through the same pipe and washes the cuttings to the surface. The pipe is lengthened as the well goes deeper, and at the end of drilling the pipe column may be 1.5 to 2 kilometers long, or even longer.

The rotary drilling method has a number of disadvantages caused by the considerable length of the rotating pipe column. This makes it necessary to use a large amount of energy to move (rotate) so long a column.

Turbine Drilling

During recent years a new drilling method -- turbine drilling -- has been introduced.

Turbine drilling was invented by the Soviet engineer Kapelyushnikov, and modified by engineers Shumilov, Ioannesyan and others.

A small turbine -- the turbine drill -- with the bit attached to it is lowered into the well and is actuated by a clay colution, which behaves in the same way as during rotary drilling. However, in this case the solution also has an additional function, namely, it acts as the motive force for driving the turbine drill.

The advantages of turbine drilling are in that the pipes are motionless and therefore energy is not required to turn them, the drilling is noiseless, and pipe breakage is almost completely eliminated.

Extraction

There are three methods of petroleum extraction: gusher,

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compressor and deep pumping.

In gusher extraction, the oil is raised to the casing head by the pressure of gas or water, then passes through the Christmas tree and pipes into a tank. The pressure in the well is regulated in such a way as to avoid the useless escape of gas into the atmosphere, so that all of its energy is directed only to lifting the oil to the earth's surface.

In compressor extraction the pressure in the producing horizon gradually weakens, and at length the moment arrives when it is only enough to lift the oil to a certain height in the well, without bringing it all the way to the surface. At this time, gas (gas lift) or air (air lift) is then employed to lift the oil to the surface. The gas or air is pumped into the well by compressors, and this method of extraction is therefore termed the compressor method.

In deep-pumping extraction the pressure in the producing horizon finally becomes so weak that the oil is lifted only a short distance above the well bottom. A special pump adapted for pumping oil from great depths is then lowered into the well.

The compressor and deep-pumping methods of extraction are also called the mechanized methods.

The sequence of extraction methods we have indicated is not obligatory. There may be cases of drilling into old and already exploited strata, and extraction is thus commenced with the compressor method or even with the deep-pumping method.

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THE GATHERING OF OIL ON THE PRODUCING LOTS AND ITS TRANSPORTATION TO THE REFINERIES

Oil extracted on the producing lots is gathered in large metal tanks. It must be processed and therefore must be transported to the refineries.

There are a number of methods of transporting oil from wells to refineries.

The most convenient method is pumping it through pipe lines. Although this method is compulsory for wells located near the refinery, there are also long pipe lines.

During the navigation season oil is shipped to plants at great distances from its place of extraction in special oil barges. Such plants ordinarily have a sufficient supply of raw material (petroleum or mazut) shipped to them to assure their operation until the following navigation season.

In some cases the raw material must be transshipped in railroad tank cars, but this is the most expensive and disadvantageous method of shipping petroleum to the refineries.

CHAPTER ONE

THE PHYSICAL PROPERTIES OF PETROLEUM AND PETROLEUM PRODUCTS

1. The Specific Gravity of Petroleum Products

The ratio of a given volume of a petroleum product at a temperature of 20 degrees C to the weight of the same volume of

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water at 4 degrees C is called its specific gravity.

The specific gravity is designated by the sign d_4^{20}, where the figure 20 denotes the temperature of the petroleum product and the figure 4, the temperature of the water.

Petroleum products expand and become lighter on heating, which means that the same volume of a petroleum product weighs less at a higher temperature than it does at a lower temperature. Consequently its specific gravity decreases with increasing temperature.

If the specific gravity of a petroleum product at any given temperature t is known, its specific gravity at 20 degrees C may be calculated by the formula:

$$d_4^{20} = d_4^{t} + \gamma (t-20)$$

where γ is the temperature correction determined from the table of temperature corrections (Table 2).

Table 2

Table of Temperature Corrections

Limits of Specific gravities	Temperature Correction for 1 degree	Limits of Specific gravities	Temperature Correction for 1 degree
[1]	[2]	[3]	[4]
0.700 - 0.710	0.000897	0.850 -0.860	0.000699
0.710 - 0.720	0.000884	0.860 - 0.870	0.000686

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[1]	[2]	[3]	[4]
0.720 - 0.730	0.000870	0.870 - 0.880	0.000673
0.730 - 0.740	0.000857	0.880 - 0.890	0.000660
0.740 - 0.750	0.000840	0.890 - 0.900	0.000647
0.750 - 0.760	0.000831	0.900 - 0.910	0.000633
0.760 - 0.770	0.000818	0.910 - 0.920	0.000620
0.770 - 0.780	0.000805	0.920 - 0.930	0.000607
0.780 - 0.790	0.000792	0.930 - 0.940	0.000594
0.790 - 0.800	0.000778	0.940 - 0.950	0.000581
0.800 - 0.810	0.000765	0.950 - 0.960	0.000567
0.810 - 0.820	0.000752	0.960 - 0.970	0.000554
0.820 - 0.830	0.000738	0.970 - 0.980	0.000541
0.830 - 0.840	0.000725	0.980 - 0.990	0.000528
0.840 - 0.850	0.000712	0.990 - 1.000	0.000515

For instance, the specific gravity of a product at 50 degrees C is 0.835. Required, to find the specific gravity of this product at 20 degrees C. The temperature correction 0.000725 corresponds to the specific gravity 0.835. We substitute this value in formula (1):

$$d_4^{20} = 0.835 + 0.000725\ (50-20) = 0.835 + 0.0217 = 0.8567.$$

Methods of Determining Gravity

The specific gravity of petroleum products is ordinarily determined in the laboratory by the following three methods: hydrometer, Westphal balance and pycnometer.

The simplest method (but also the most inexact) is determina-

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tion by the use of the hydrometer. The hydrometer is a cylindrical glass vessel (Figure 5) with a bulb at the bottom filled with mercury. The lower wide part of the apparatus is a thermometer. The upper narrow part of the hydrometer has graduations every 0.0005 (specific gravity) in terms of the density of water at + 4 degrees C.

Table 3

Table of Hydrometer Graduations

Products	Graduations of Specific Gravity
For light gasolines	0.648 - 0.712
For heavy gasolines	0.708 - 0.772
For ligroins and kerosenes	0.768 - 0.832
For gas oil, solar and light oils	0.828 - 0.892
For heavy petroleum products	0.888 - 0.952

The products are poured into a glass or metal cylinder with a diameter of at least 5 cm. A clean and dry hydrometer is lowered into the liquid, and after the oscillations have ceased a reading is taken at the upper meniscus of the petroleum product. At the same time the temperature of the product is determined.

Let us assume that a reading is 0.835 at temperature 50 degrees C. As we have seen above, the specific gravity at 20 degrees C is 0.8567. The specific gravity of viscous petroleum products with viscosity higher than $E_{50} = 25$ is determined after their dilution with an equal volume of tractor kerosene with known specific gravity.

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The specific gravity of this mixture is equal to the sum of the specific gravity of the kerosene and the specific gravity of the petroleum product being examined, divided by 2.

Let d_1 be the gravity of the mixture, d_2 the gravity of kerosene, and d the gravity of the petroleum product.

Then

$$d_1 = \frac{d_2 + d}{2}$$

Whence

$$2d_1 = d_2 + d.$$

Solving the equation for d:

$$d = 2d_1 - d_2.$$

The specific gravity of the mixture was found to be 0.0895.

The specific gravity of tractor kerosene is commonly known to be 0.825. Substituting the known values in formula (2), we obtain

$$d = 2 \cdot 0.895 - 0.825 = 0.965.$$

The Westphal balance (Figure 6) consists of the stand 1-2 with the adjusting screw 3, the prism 4, the balance beam 5, a glass float and a set of small rider weights. At one arm of the balance beam there is a counterpoise while on the other arm, graduated into ten divisions, a glass float is suspended. The weights are so adjusted that, when the float is not immersed in liquid and there are no rider weights on the other arm, the balance is in equilibrium. When the float is immersed in distilled water at + 4 degrees C, the equilibrium is disturbed, since the float displaces water in an amount equal to its volume. To restore equilibrium a rider must

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be placed at division 10 with a weight equal to that of the displaced water. There is a second rider 0.1 the weight of the first, a third one 0.01 that weight, and a third one 0.001 that weight.

Specific gravity is determined as follows on the Westphal balance. The float is immersed in the petroleum product and the equilibrium is consequently disturbed. Riders are placed on the divisions until equilibrium is restored, and then the readings are taken. Assume that the first rider is at division 8, the second at division 5, the third at division 2 and the fourth at division 5. The specific gravity at the temperature of the liquid will be 0.8525. The calculation of d_4^{20} is made in the same way as with the hydrometer.

The method of determining the specific gravity with a pycnometer is very exact. It requires very careful work and is performed only by qualified workers. Pycnometers may have a volume of 1 to 25 c.c. (Figure 7). They are weighed on the very accurate so-called analytical balance.

The specific gravity of the various crudes and petroleum products. The gravity of crude oils varies over a very wide range, from 0.750 to 1.000. For instance, the specific gravity of Baku crudes ranges from 0.780 to 0.930, that of Sakhalin crudes from 0.830 to 0.940.

The specific gravity of the various products obtained from crude oil likewise varies greatly: light, bright petroleum products have low specific gravities, while dark products have high specific gravities. The specific gravity of gasoline is not over

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0.760, that of ligroin or petroleum ether not over 0.795, that of kerosene not over 0.830, that of gas oil not over 0.875, that of solar oil not over 0.880. The specific gravity of light lubricating oils is between 0.860 and 0.900, and that of heavy lubricating oils runs to 0.930. The specific gravity of mazut may run as high as 1.

2. The Viscosity of Petroleum Products

When a liquid flows a resistance develops inside it which interferes with the mutual motion of the particles of the liquid. This resistance is termed internal friction or viscosity.

Relative Viscosity

In the work of plants and commercial oil bases the so-called relative viscosity is used, showing how many times the viscosity of the liquid in question at a certain temperature is greater or smaller than the viscosity of water at 20 degrees C.

In the USSR the relative viscosity is determined by the Engler viscosimeter. Viscosities determined with this apparatus are expressed in Engler degrees, abbreviated $^{o}E_{t^{o}C}$.

The ratio of the time in seconds required for 200 milliliters of the product tested to flow out of the apparatus, at a given temperature, to the time required for 200 milliliters of distilled water to flow out of it at 20 degrees C is termed the viscosity (in Engler degrees). The viscosity is generally determined at 50 or 100 degrees C.

The Engler viscosimeter (Figure 8) consists of two brass reservoirs: the internal reservoir 1 for the petroleum product

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and the external reservoir 2 for water or oil, by which the petroleum product is heated. There is an aperture in the center of the reservoir from which the liquid may flow. A brass tube containing a small platinum tube is soldered into the aperture. The opening is closed by the wooden plunger b.

The viscosity at 50 degrees C is determined in the following manner. The petroleum product is heated to 52 degrees C and poured into the apparatus up to the mark. The outer reservoir is filled with water at 52-53 degrees C. The temperature of the petroleum product is then brought to exactly 50 degrees C and so regulated that it does not change during the entire time of determining the viscosity. The plunger is then withdrawn and a stopwatch started. When the oil in the measuring flask 7 has reached the 200 milliliter mark, the stopwatch is stopped. Assume that the oil takes 3 minutes 55.6 seconds (or 235.6 seconds) to flow.

To compute the relative viscosity, the time of flow in seconds must be divided by the time of flow of the same quantity of water at 20 degrees C. The latter value will be constant for each apparatus and is roughly equal to 49-51 seconds. Let us assume that the water number of our apparatus is 50.5 seconds. We find the value for the viscosity of the petroleum product:

$$^{\circ}E_{50} = \frac{235.6}{50.5} = 4.66.$$

Dependence of Viscosity on Temperature

As the temperature is increased the viscosity of all petroleum products falls, and, on the other hand, as the temperature falls

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the viscosity rises. We present the variation with temperature of the viscosity of a few petroleum products and of vegetable colza oil in the form of curves (Figure 9). The temperatures are plotted on the horizontal axis, and the viscosity in $^{\circ}E_{t^{\circ}C}$ on the vertical. As may be seen from curve 4, the viscosity of lubricating oil at 10 degrees C is 80, at 20 degrees C it falls sharply to 30, and at 30 degrees C it is 17 degrees E.; beyong this point it declines slowly with increasing temperature. The viscosity of cylinder oil (curve 2) likewise varies sharply with changing temperature; it is 80 at 50 degrees C, 34 at 60 degrees C, and 20 at 70 degrees C; beyond this point it changes only slowly.

Such petroleum products are said to have high viscosity curves. Their use is very inconvenient: at low temperatures, i.e., under the conditions of their transfer and of the starting of the motor, they are very viscous, but under operating conditions, when the motor is running at increased temperatures, they become thinned down and have so slight a viscosity that they cannot assure normal operation of the frictional parts of the motor.

Substances with a flat viscosity curve are the best. For instance, colza oil (curve 5) has a viscosity of 16 at 15 degrees C, of 8 at 30 degrees C, of 6 at 40 degrees C, of 4 at 50 degrees C, and so on. Transformer oil (curve 6) also has a flat viscosity curve.

Such a course of variation in viscosity is the most valuable property of lubricating oils. Oils with flat viscosity curves behave considerably better in operation than do oils with high viscosity curves.

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At the present time it is customary to judge the quality of a lubricating oil by a conventional value called the index of viscosity. Oils with the higher indices of viscosity (100 and higher) are the best. Truck engines require oils with indexes of viscosity from 65 to 75, while airplane engines must operate on oils with indexes of viscosity of 100 and above.

3. Flash point, Fire point, and Spontaneous-ignition Point

When petroleum and petroleum products are heated, vapors of the lighter fractions are given off. The higher the temperature to which the product is heated, the more vapor is released. According to the quantity of vapors given off, a petroleum product may give a flash when fire is applied, or may itself take fire. At a high enough temperature the petroleum product may ignite even in the absence of fire. This phenomenon is termed spontaneous ignition.

The Flash point

The temperature at which the vapors of a petroleum product, mixed with air, give a short flash under certain conditions (on the application of a flame) is called the flash point. The flash flame goes out and the liquid itself does not take fire. The flash-point characterizes the fire hazard of a petroleum product and the presence of lighter components in it.

Gasoline gives off so great an amount of vapor that it gives a flash at temperatures considerably below 0 degrees C.; this indicates the great fire hazard of gasoline.

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Kerosene gives a flash only when it is heated. At a temperature of 28-30 degrees C, sometimes even higher, a sufficient quantity of kerosene vapor is formed to flash on the application of a flame.

The heavier petroleum products need even more heating than this. For instance, some lubricating oils have flash points over 300 degrees C.

There are many factors that influence the flash point of a petroleum product: the shape of the apparatus, the quantity of liquid heated, the size of the flame, the rapidity of heating, and so on. The flash point is therefore determined in special apparatus under certain standard conditions.

In the USSR the flash-point is customarily determined in apparatus of two types: in a closed tester (Pensky-Martens, Figure 10a) and in the open-cup tester (Brenken, Figure 10b).

In the Martens-Pensky tester the petroleum product is heated in a small metal reservoir at a rate of 5 - 8 degrees C per minute. At a temperature 10 degrees C below the expected flash point, the lid of the apparatus is opened and a small lighted lamp is simultaneously applied to the opened orifice. If a sufficient quantity of vapor has already been formed, it will flash on application of the lamp. This then will be the flash-point. If a flash does not occur the lid is opened with every 2 degrees C rise in temperature until the vapors do flash.

The Brenken apparatus is much simpler. It consists of a small iron crucible placed in a sand bath. The crucible is filled with the product up to a certain level, a thermometer inserted,

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and the product is heated at the rate of 10 degrees C per minute. At a temperature 40 degrees C below the expected flash point the heating is slowed down to 4 degrees C per minute. From 10 degrees C below the expected flash point a flame is applied to the apparatus with every 2 degrees C rise in temperature until the vapors finally flash.

The Brenken apparatus is used to determine the flash points of heavy petroleum products.

The flash point of one and the same petroleum product have different values when determined by Martens-Pensky and Brenken testers (Table 4).

TABLE 4

	Brenken tester degrees C	Martens-Pensky tester degrees C	Difference degrees C
Crude oil	46	30	16
Mazut	119	96	23
Solar oil	160	148	12
Machine oil	200	180	20
Machine oil with 0.5% gasoline	200	80	120
Cylinder oil	236	215	21

Flash points determined by the Brenken tester are always somewhat higher. This is explained by the fact that, in the open cup, part of the vapor given off on heating escapes into the atmosphere, thus delaying the flash. The difference between the flash points as

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determined by the Brenken and Martens-Pensky testers is particularly great for such varied products as mazut, crude oil and lubricating oil contaminated with small amounts of gasoline. Thus, the flash point of lubricating oils may be used as an index of their uniformity and purity, which is very important.

The flash point determined in the open-cup tester is conventionally designated as: Brenken flash point. The flash point determined in the closed tester is called: M. P. flash point.

Fire Point

When a product is heated above its flash point a time is reached when the liquid itself is ignited. If beore ignition the liquid burns for not less than 5 seconds, the corresponding temperature is called the burning point. The lower the burning point, the greater the fire hazard of the petroleum product.

Spontaneous-ignition point

The temperature at which a mixture of vapors and air (heated to a high temperature) ignites without the application of a flame is called the spontaneous-ignition point of a petroleum product, and may sometimes differ from the flash point by several hundred degrees. For instance, the flash point of gasoline is 15-20 degrees C below zero, while its spontaneous-ignition point is 400 degrees C. This difference is considerably less for heavy, viscous products.

The spontaneous-ignition point is important for Diesel engines, in which the fuel is ignited on mixture with compressed air heated to a high temperature.

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4. Melting and Freezing of Petroleum Products

Substances like water or benzol, for instance, have a definite, constant melting point: water freezes and ice melts at 0 degrees C, benzol at + 5 degrees C.

Crude oil and petroleum products are complex substances, consisting of mixtures of various hydrocarbons, liquid and solid, of tars, asphalts and other substances. For this reason the transition of a petroleum product from a liquid state to a solid, i.e., its congelation or freezing, and its transition from the solid state to the liquid, i.e., its melting, does not occur all at once at any single definite temperature. Petroleum products and crude oil, before congealing, pass through an intermediate stage of gradual thickening within the limits of several degrees. Melting also does not occur all at once but is accompanied by a fairly protracted stage of softening. These stages of thickening and softening (meaning freezing and melting) of petroleum products depend on the methods by which they are determined (on the apparatus, quantity of the substance, rate of heating, etc).

The standard method of determining the freezing point of petroleum products is by using a test tube 15-17 millimeters in diameter, filled to a height of 3 centimeter with the product, in which a thermometer is immersed. (Figure 11). The test tube and contents are first heated to 50 degrees C, then chilled for 5 minutes in a chilling mixture at a temperature of 2 - 3 degrees C below the freezing point of the product in question. The test tube is placed in an inclined position (at an angle of 45 degrees)

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and allowed to stand for another five minutes. If the surface of the liquid does not move, this means that the product is frozen. If the surface of the liquid does move, however, the procedure is repeated at a temperature 2 degrees C lower.

Thus, the freezing point may be defined as the temperature at which a petroleum product loses its mobility under certain standard conditions.

The freezing point determined by this method is of a relative and conventional nature. It does not in fact correspond to the actual solidification temperature of most crude oil or petroleum products, which retain their mobility at temperatures considerably lower than their freezing points as determined by the laboratory method. The laboratory data make it possible to compare various petroleum products according to their ability to solidify under identical conditions.

The freezing point of a product depends on its content of paraffin: the more paraffin it contains, the quicker does it freeze. Thus, for instance, Grozniy paraffin crude freezes at + 12 degrees C, while the mazut obtained from it freezes at + 36 degrees C, because the mazut contains more paraffin than the original crude. Emba, Baku, and also the Grozniy paraffin-free crudes, and the mazut prepared from them, do not lose their mobility at temperatures of -- 20 degrees C.

The practical significance of the freezing point in the operation of plants and petroleum bases is very great. The method of processing a crude can be worked out sooner if we know its freezing

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point, and the necessary measures can be taken for its transfer by pumping during the cold season of the year, and methods of storage and shipment of high freezing-point petroleum products can be selected.

5. The Color of Petroleum Products

Crudes from different deposits and regions may differ in color: the color of crude oil varies within a very wide range, from light-colored, almost colorless, to completely black. Crude oil may be light or dark yellow, light or dark red, light or dark brown.

The white crude of Surakhan, which is no longer longer produced, was a colorless crude. The color of a crude depends on its composition and its content of tarry and asphaltic substances. The higher the content of gasoline-kerosene fractions and the lower the content of tarry substances, the lighter the color of a crude.

The above-mentioned Surakhan white crude oil had a gravity of 0.738 and consisted entirely of gasoline-kerosene fractions, with no tar at all. Another Surakhan crude -- the red -- has a specific gravity of 0.810, and consists of 85 percent gasoline-kerosene fractions. Heavy tarry crudes are black and have gravities close to 1.000. Thus, a definite connection is observed between the color of a crude and its gravity: the heavier the crude, the darker it is. The color of petroleum products obeys the same rule.

The color of kerosene and that of lubricating oils is

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determined in the laboratory by instruments called colorimeters (Figure 12). In these devices the color of a layer of the petroleum product is compared with that of standard solutions or of colored glasses. The color of kerosene as determined by a colorimeter is expressed in millimeters of a kerosene layer of which the color coincides with that of the standard glasses. The thicker the layer, the higher the quality of the kerosene.

The color of lubricating oils is also determined in a standard instrument and is expressed in millimeters of a column of a standard solution the color of which coincides with the color of the oil.

The color of petroleum products is an index of their purity and of the absence of tarry and colored substances.

CHAPTER TWO

THE THERMAL PROPERTIES AND FRACTIONAL COMPOSITION OF CRUDE OIL [redacted] AND PETROLEUM PRODUCTS

6. Evaporation and boiling of pure substances

We often observe vapor rising from the surface of water in consequence of its evaporation. This phenomenon may be seen both in summer and in winter, at any temperature.

Evaporation takes place only from the surface of a liquid. A considerable number of causes affect the rate of evaporation: the temperature of the liquid, the area of the surface, the depth of the layer of liquid and the rate of motion of the air.

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Let us perform the following experiment: we heat an open vessel containing water. The higher the temperature to which the water is heated, the more rapidly the water will evaporate. The more vapor above its surface the higher the vapor pressure. At last the vapor pressure of the liquid equals the atmospheric pressure. Evaporation will then take place not only from the surface but from the whole mass of the water. In spite of the uninterrupted heating of the water, its temperature will not rise further. If the outside pressure is normal, that is, equal to 760 millimeters of mercury, the temperature of the water will be 100 degrees C. The phenomenon we have described is termed boiling.

Boiling differs from evaporation in that: (a) evaporation occurs only from the surface, while boiling involves the entire mass of liquid; (b) evaporation occurs at any temperature, while boiling takes place only at one temperature which is constant for a given liquid. For instance, the boiling point of water under normal pressure is 100 degrees C, for benzol it is 80.4 degrees C, for ethyl alcohol it is 78 degrees C.

In boiling, the external pressure is overcome by the pressure (or tension) of the vapor of the liquid. If the external pressure is increased, for instance, to 2 atmospheres (1520 millimeters of mercury), then water will not boil at 100 degrees C, since at that temperature the pressure (or its vapor tension) is 760 millimeters, while a vapor tension equal to 1520 millimeters of mercury is required to overcome the external pressure.

To overcome an atmospheric pressure increased to 1520 millimeters, water must be heated to the temperature at which its vapor

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pressure will equal 1520 millimeters. It is well known that this temperature is 121 degrees C. At a pressure of 3 atmospheres the boiling point of water is 134 degrees C, at 4 atmospheres it is 144 degrees, at 5 atmospheres 153 degrees, at 10 atmospheres 181 degrees, etc.

The higher the temperature, the more rapidly the particles of the liquid move and the more easily they tear away from the surface of the liquid and pass into the vapor space, or, as we say, into the vapor phase (water in the liquid state being in the liquid phase). At the boiling point of a substance, as we have already learned, the vapor tension is equal to the external pressure. The vapor phase is completely saturated by the vapor of the liquid, and the temperatures of the vapor and the liquid are the same. In this case, along with the evaporation of the liquid, part of the vapor will be condensed and pass back into the liquid and a state of equilibrium between liquid and vapor will be attained. Such vapor is termed saturated, and its tension is the tension of saturated vapor. The tension of saturated vapor is constant for each substance. For instance, at 100 degrees C the tension of saturated water vapor is 760 millimeters of mercury, at 134 degrees C it is 2280 millimeters (3 atmospheres), at 181 degrees C it is 7866 millimeters (about 10 atmospheres).

The saturation vapor pressure is strictly dependent on the temperature of the liquid: the higher this temperature, the higher the saturation vapor pressure.

Oil refineries often must deal with superheated or un-

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saturated vapor, which does not obey this rule. The tension of superheated vapor is lower than that of saturated vapor at the same temperature. In other words, superheated vapor occupies a greater volume than saturated vapor does. Superheated vapor may be converted into saturated vapor by gradually compressing it to the same volume occupied by saturated vapor.

7. Evaporation and Boiling of Crudes and Petroleum Products. The fractional Composition of Petroleum Products

The evaporation and boiling of such complex substances as crude oil and petroleum products differs from the evaporation and boiling of simple substances.

Crude oil and petroleum products consist of mixtures of various classes of hydrocarbons, which differ from each other in gravity, boiling point and other properties.

When crude oil or petroleum products are heated, no definite boiling point is observed as is the case when any simple substance is heated. If the temperature of the products being heated is steadily increased, they will evaporate continuously (beginning with the lightest components) until evaporation is complete. Consequently, we cannot speak of any definite boiling point of a petroleum product, but must instead speak of the temperatures at which this or that proportion of the product will have boiled away. This determination is made in a standard Engler laboratory still (Figure 13).

The still consists of the flask 1 (of definite size), the

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condenser 3, the receiver 4, the outer shell 5, the thermometer 2 and the burner 6. Let us fill the flask with water, place the thermometer in it, and start heating it. At 100 degrees C the water will boil; the water vapor passes through the discharge vent into the condenser, is condensed there and drains into the receiver. Up to the very end of boiling the thermometer will continue to read 100 degrees C until all the water has been evaporated.

This process is termed distillation. The product collected in the condenser is termed condensate or distillate. The condensate from the distillation of water is distilled water.

When petroleum or petroleum products are heated, distillation also takes place, but the character of that distillation is entirely different. The distillation of a pure substance takes place at one and the same temperature, which is constant for the given substance, while the distillation of petroleum products takes place at various gradually increasing temperatures.

Let us analyze this in an example. We charge the flask of the still with 100 cc of motor gasoline and heat it. In a few minutes the first drop of the product drips into the receiver. The temperature at which the first drop of product passes over is termed by general agreement the initial boiling point.

Assume that the first drop of product passes over at temperature 50 degrees C., the initial boiling point (NK) will consequently be 50 degrees C. After this the drops of product will continue to fall without interruption, at a certain definite speed. At each 10 degrees C we change the receivers. At 60 degrees C we note what

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volume of liquid has condensed, let us say 2 cc. This amount is the temperature fraction (or simply the fraction) boiling at temperatures between 50 and 60 degrees C. In exactly the same way we note the volume of the distillate passing over up to 70 degrees C, up to 80 degrees, etc. Since the flask has been charged with 100 cc, the number of cc distilled with each fraction show the percentages of such fractions.

The term end point is conventionally applied to that maximum temperature at which the entire product finally passes over into the vapor state. The end point (KK) for gasoline corresponds to the moment at which 97.5 percent -- 98 percent of the entire product has passed over. The losses and the residue in the flask after distillation amount together to 2 - 2.5 percent. The fractions distilled at definite temperatures characterize the fractional composition of petroleum products.

The fractional composition of crudes and petroleum products may be expressed by means of a curve. For this purpose the percentages of the fraction distilled are plotted along the horizontal axis, and the boiling points of these fractions are plotted along the vertical axis. A curve is then drawn through the points so obtained.

In the distillation of gasoline it may also be noted at what temperature 10 percent, 20 percent, etc, of the product is distilled.

Let us construct the curve for motor gasoline (see Figure 14),

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having the following composition:

Initial Boiling Point	50 degrees C
10% passes over at	73 degrees C
20% passesover at	91 degrees C
30% passes over at	103 degrees C
40% passes over at	115 degrees C
50% passes over at	125 degrees C
60% passes over at	137 degrees C
70% passes over at	148 degrees C
80% passes over at	163 degrees C
90% passes over at	185 degrees C
98% (end point)	210 degrees C

To note all these points at each distillation of a petroleum product would be a complicated task, and there is no need for doing it. For this reason 3 to 4 of the most characteristic points for each petroleum product have been adopted by general agreement for evaluating its fractional composition. For instance, aviation gasoline must have the following fractional composition:

Initial boiling point	40-75 degrees C
10% passes over at a temperature not lower than	80 degrees C
50% passes over at a temperature not lower than	112 degrees C
90% passes over at a temperature not lower than	150 degrees C
End point not above	180 degrees C
	(97.5%).

The fractional composition of petroleum products is one of the most important indices of their quality.

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8. Thermal Capacity and Enthalpy of Petroleum Products. Latent Heat of Evaporation

To heat the same quantity of different substances requires different amounts of heat. The amount of heat required to raise the temperature of 1 gram of a substance by 1 degree C is termed its thermal capacity. The calorie, taken as the unit of thermal capacity, is the quantity of heat required to raise the temperature of 1 gram of distilled water 1 degree C, from 19.5 to 20.5 degrees C at normal pressure. This is the small calorie (abbreviated cal). The large calorie (Cal) is equal to 1,000 small calories. (kilocalorie or kcal). The unit of thermal capacity, the calorie, is fixed for a definite temperature (19.5 to 20.5 degrees) because the thermal capacity of substances increases with rising temperatures.

The thermal capacity of a substance at any temperature is called its true thermal capacity. For instance the true thermal capacity of kerosene, of specific gravity d = 0.825, at 38 degrees C is 0.47 Cal/kg, and at 100 degrees C it is 0.53 Cal/kg.

In computations the so-called mean thermal capacity is used. This is equal to one-half the sum of the true thermal capacities at the initial temperature and the final temperature. For instance, the mean thermal capacity for kerosene on heating from 38 to 100 degrees C is equal to

$$\frac{0.47 + 0.53}{2} = 0.5 \text{ Cal/kg.}$$

Let us calculate how much heat is required to heat 10 kg of kerosene from 38 to 100 degrees C.

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We use the formula

$$Q = g\, C_{mean}\, (t_2 - t_1),$$

where g is the quantity of substance to be heated,

t_2 is the final temperature,

t_1 is the initial temperature,

C_{mean} is the mean thermal capacity.

$$Q = 10 \cdot 0.5(100 - 38) = 360 \text{ Cal/kg.}$$

Each kilogram of kerosene receives 36 large calories. This is the so-called increment of enthalpy, i.e., the amount of heat necessary to heat 1 kg of kerosene from 38 to 100 degrees C.

Enthalpy, or heat content, is the quantity of heat required to heat 1 kg of a substance from 0 degrees to t degrees C. Then the enthalpy at any given temperature t degrees C of a substance is the supply of heat obtained on heating it from 0 degrees to t degrees C without evaporation. Constancy of physical state is a necessary condition here, i.e., up to the end of the heating the substance must be in the liquid, solid or gaseous state.

The thermal capacity of a petroleum product and consequently its enthalpy, depend on its specific gravity: the higher the specific gravity of the product, the lower the thermal capacity. For instance, at 20 degrees C:

a petroleum product with specific gravity d =

0.700	has a thermal capacity of 0.50
0.750	0.48
0.800	0.46
0.850	0.45
0.900	0.44

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The enthalpy of a saturated vapor is the amount of heat required to heat 1 kg of the substance from 0 degrees C to the boiling point, plus the latent heat of vaporization of the substance.

The latent heat of vaporization of a substance is the quantity of heat required to transform 1 kg of the substance into saturated vapor at the boiling point under normal atmospheric pressure.

The following mean values are taken for the latent heat of vaporization of petroleum products under atmospheric pressure:

Gasoline	70 Cal/kg	Solar and spindle distillate	50 Cal/kg
Ligroin	65 Cal/kg	Paraffin distillate	47 Cal/kg
Kerosene	60 Cal/kg	Machine-oil distillate	45 Cal/kg
Gas oil	55 Cal/kg	Cylinder-oil distillate	40 Cal/kg

As is apparent, the latent heat of vaporization of a petroleum product declines with increasing heaviness of the product.

The enthalpy of saturated vapors of petroleum products varies within a range of 130 - 190 Cal/kg. The enthalpy of superheated vapor is higher than that of saturated vapor by the quantity of heat necessary to bring the vapor from the boiling point to the superheat temperature.

9. The Heat Value of Petroleum Products

The quality of petroleum products as fuels is primarily evaluated by their heat value, i.e., by the quantity of heat given off by 1 kg (or 1 cubic meter, for gases) of the product on complete combustion.

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If the products of combustion are cooled to the temperature of the surrounding medium, the maximum quantity of heat is given off. This quantity of heat is termed the maximum heat value of a fuel -- Q maximum (Q_{max}).

In practice it is unnecessary to cool the products of combustion to such an extent. The minimum heat value of a fuel -- Q minimum (Q_{min}) or Q working (Q_{work}) is therefore usually employed. These values are less than Q_{max} by the amount of the heat of vaporization of the water contained in the products of combustion.

The heat value of a fuel may be determined very exactly by burning a definite amount of it in a special apparatus called a "bomb". If the ultimate analysis of the fuel is known, that is, the percentage of carbon, hydrogen and other elements contained in it, its heat value may be determined by Mendeleyev's formula:

$$Q_{work} = 81C + 246\ H - 26\ (O - S) - 6W,$$

where C, H, O, S and W are the respective percentages of carbon, hydrogen, oxygen, sulfur and water in the fuel.

The heat values of a few petroleum products are given below.

	Cal/kg		Cal/kg
Gasoline	11230	Mazut	10843
Kerosene	11059	Balakhan crude	10590
Solar oil	10914	Binagad crude	10430
Spindle oil	10894	Natural gas (Cal/cubic meter)	8000
Machine oil	10899	Manufactured gas (Cal/cubic meter)	9000 - 10000
Cylinder oil	10883		

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CHAPTER THREE

THE CHEMICAL COMPOSITION, PROPERTIES AND CLASSIFICATION OF CRUDES

10. The Concept of the Group Chemical Composition of Petroleum Products

Crude oil, as everyone knows, represents a very complex mixture of hydrocarbons of various classes, oxygen, sulfur and nitrogen compounds, and also tarry and asphaltic substances.

In consequence of the immense diversity of the hydrocarbons, determination of the exact chemical composition of petroleum or of any petroleum product is a very complicated task. Analysis is therefore confined to determination of what is called the group chemical composition, namely, the quantities not of the separate hydrocarbons contained in a crude or petroleum product but of their groups or classes.

The chemical composition of petroleum and petroleum products affects their chemical and physical properties. For instance the predominance of paraffin hydrocarbons in a crude, and consequently of heavy paraffin, makes its freezing point high under ordinary conditions and its mobility low.

Let us consider the properties of the separate groups of hydrocarbons and certain other compounds that enter into the composition of crude.

11. The Hydrocarbons of Petroleum

The paraffin hydrocarbons of petroleum are the most widely

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distributed class of hydrocarbons in petroleum: all natural gases consist of paraffin hydrocarbons. Gasolines consist chiefly of liquid members of this class. Other petroleum products also contain high percentages of paraffin hydrocarbons; the heavy paraffins consist entirely of paraffin hydrocarbons. The gasolines yielded by most crudes contain 40-60 percent paraffin hydrocarbons. Gasolines from naphthene-base crudes have 25-35 percent of these hydrocarbons, while they run up to 70 percent in gasolines from paraffin-base crudes. In kerosenes they range from 10 to 60 percent.

The liquid paraffin hydrocarbons play an immense role as the chief constituents of the gasolines. For engine operation, a principal characteristic of gasolines is their anti-knock quality, which is determined by their octane number (see 13). Paraffin hydrocarbons of normal structure have the lowest octane numbers, while those of isomeric structure have high octane numbers.

The solid paraffin hydrocarbons, beginning with $C_{16}H_{34}$ and running up, are components of various crudes.

Technical paraffin consists of representative of this group and is prepared from paraffinic crudes. The paraffin content of crudes varies within a very wide range, from fractions of a percent to 6 - 8 percent. Occasional crudes with over 10 percent paraffin are known.

Crudes containing paraffin in considerable amounts solidify at high temperatures (at 0 degrees C and higher), which must be kept in mind in choosing the method of storage, shipment and processing of such crudes.

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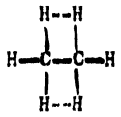
As may be seen from the formulae of the paraffin hydrocarbons, they are saturated hydrocarbons, i.e., all bonds of the carbon atoms are completely saturated:

Methane

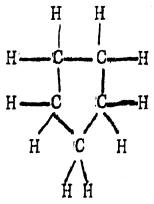


Ethane

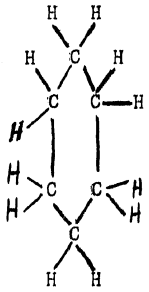
The paraffin hydrocarbons are therefore chemically stable and are not capable of addition reactions.

In other words these hydrocarbons are called saturated or methane hydrocarbons.

The naphthene hydrocarbons of petroleum, the main constituents of petroleum, are a second class of hydrocarbons. In their chemical properties the naphthenes recall the paraffin hydrocarbons. Their chemical activity is also low. But they are constructed in a completely different manner: they are of closed-ring (cyclic) character. For this reason the naphthene hydrocarbons are also called cycloparaffins.



Cyclopentane



Cyclohexane

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Naphthenes of only two series, cyclopentane and cyclohexane, are contained in petroleum. The members of these series are distinguished from each other by the presence of various side chains, for instance: methylcyclopentane, dimethylcyclopentane, ethylcyclopentane, etc.

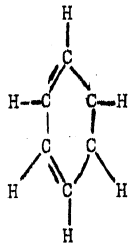
There is great variety observed in the content of the naphthene hydrocarbons in different crudes. The Baku, Emba, and Grozniy paraffin-free crudes are richest in such hydrocarbons.

As constituents of gasolines and kerosenes the naphthene hydrocarbons are of great importance on account of their high anti-knock properties.

They are also important in lubricating oils because of their low freezing points and high lubricant qualities.

Aromatic hydrocarbons, in comparison with the paraffin and naphthene hydrocarbons, are present in smaller quantities in crudes; while gasolines generally contain no more than 10 percent, and kerosenes from 15 to 25 percent.

Benzol is the first representative of the aromatic hydrocarbons:



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The aromatic hydrocarbons have high antiknock properties and therefore play a great part as constituents of gasolines and kerosenes.

In some cases the aromatic hydrocarbons are useless and even harmful. They are harmful, for instance, in special "galosh" gasoline on account of the higher toxicity of their vapors. They are harmful in illuminant kerosenes, since they give soot and deposits of carbon when burned. High-molecular aromatic hydrocarbons are also harmful in lubricating oils, since they are readily oxidized at high temperatures by the oxygen of the air and thus impair the quality of the oil. In all these cases the aromatic hydrocarbons are removed from the distillates by further refining.

Crudes do not contain unsaturated hydrocarbons. However, when crudes are refined, especially by cracking or pyrolysis (see Chapter VII), large amounts os such hydrocarbons are contained in the products so produced. Unsaturated hydrocarbons are also called olefinhydrocarbons.

Unsaturated hydrocarbons are unstable substances and readily resinify in air. For this reason they are harmful components in most cases, for example, in straight-run aviation gasolines, in kerosenes and in lubricating oils.

Cracked gasolines consist, to the extent of almost a third, of unsaturated hydrocarbons. It would be difficult to remove them from cracked gasolines, and so great a loss of these components would make the cracking process disadvantageous. Consequently, only the most unstable unsaturated hydrocarbons, the diolefins,

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are actually removed from gasoline.

12. Other compounds in Petroleum

Naphthenic Acids

Like other acids, the naphthenic acids attack metals, and their presence in petroleum products is therefore very undesirable. At the same time the naphthenic acids and their salts are valuable substances and so their extraction from crudes is an obligatory operation.

In their liquid form the naphthenic acids are oily liquids with an unpleasant odor, with gravities ranging from 0.960 to 1.000. They are insoluble in water but rather readily soluble in sulfuric acid, while their sodium salts are readily soluble in water, foam and have wetting properties, and are thus used in soap making.

The naphthenic acid content of various crudes ranges from fractions of a percent to 2 percent. The naphthenic crudes contain the largest amount and the paraffin crudes the lowest.

The naphthenic acids pass over together with the distillates. They are named according to the distillates from which they are separated, such as kerosene naphthenic acids, gas oil naphthenic acids, solar naphthenic acids, etc. Solar and spindle distillates have the highest content of naphthenic acids (about 2 percent). This content decreases as the distillate becomes heavier.

Sulphur Compounds

The sulfur content of the crudes from the old deposits (Baku

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and Groznly) is rather moderate and does not exceed 0.4 percent. However, one of the distinctive features of the crudes from the Second Baku is their high content of sulfur and its compounds. The sulfur content of the Bashkirian crudes is particularly high (up to 4 percent). On distillation of crudes, most of the sulfur compounds remain behind in the heavy distillates and in the mazut. These compounds are also decomposed in refining processes using decomposition (cracking processes), and a considerable percentage of sulfur thus enters the cracked gas and cracked gasolines.

Hydrogen sulfide (H_2S) and mercaptan, which actively attack metals, are the most undesirable sulfur compounds in the distillates and gas. For this reason the sulfur compounds must be eliminated from the distillates by further refining.

Tarry and Asphaltic Substances in Petroleum

Next to the hydrocarbons, tarry and asphaltic substances are the most important constituent of petroleum, ranging from a few percent to tens of percent.

The simplest method of determining tarry and asphaltic substances (but at the same time the most inexact method) is the excise test, which is made as follows.

A solution of 50 cc of the crude in 100 cc of gasoline is treated with 10 cc of concentrated sulfuric acid in a vessel with a ground-glass stopper (a separating flask); the flask is energetically shaken for 3 minutes. After standing for an hour, the number of cc of the separated mixture of sulfuric acid with

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the tarry and asphaltic substances is determined. Assume that 22 cc have separated. On subtracting the original volume of sulfuric acid, we get 12 cc.

On multiplying this volume by 2, since 50 cc of crude were taken, we obtain a figure of 24 percent for the tar.

Tarry and asphaltic substances are divided into three principal groups:

(1) neutral petroleum tars (or simply tars),

(2) asphaltenes,

(3) acid tars (asphaltogenic acids).

Neutral tars, the main group of tarry and asphaltic substances, consist of viscous, semi-liquid, dark substances heavier than water. Neutral tars have strong coloring properties. Thousandths of a percent of tar are enough to color a colorless gasoline straw yellow.

Asphaltenes are dark-brown or black powders heavier than water. Their ultimate analysis is close to that of gum, but their molecular weight may reach the order of several thousand. They cannot be distilled, and break down into coke and gas on heating. Crudes contain them in small quantities, rarely over 1.5-2 percent, and usually under 1 percent.

Natural asphalts have a large content of asphaltenes. Thus, the famous Trinidad asphalt contains 37 percent asphaltenes, 23 percent gums, 30 percent oils and 10 percent asphaltenic acids and

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their derivatives.

The mineral Substances in Petroleum

To get a complete idea of all the constituents of petroleum, we must still take up water, ash, and the group of substances termed mechanical admixtures.

The mechanical admixtures include all types of impurities (dust, clay, coke) that remain on the filter when a sample of crude oil is filtered. Crude is freed from these admixtures by standing and settling. The water and the mechanical admixtures are determined in the laboratory by the gasoline method. 50 cc of the crude are mixed with 500 cc of gasoline and allowed to stand at 70 degrees C. The amount in cc of water and dirt that separates, multiplied by 2, is the total content of water and mechanical admixtures. The Dien and Stark method (Figure 15) is used to determine water. A mixture of 100 cc of the petroleum product with 100 cc of a solvent (gasoline or ligroin) is distilled, and the water distilled off is collected separately in the receiver.

Ash is formed on combustion of petroleum and represents the residue of mineral substances soluble in petroleum (for example, the salts of napthenic acids). The ash content of a crude depends on its gum content. Low-gum crudes contain 0.002 to 0.003 percent ash; tarry crudes may have as much as 0.01 to 0.02 percent.

13. The Antiknock Properties of Fuels

Airplane engines require high-grade fuel. There are a considerable number of special requirements for aviation fuels, of

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which the principal and decisive ones are antiknock properties.

To explain what the antiknock properties of a fuel are, some knowledge of the working of an internal combustion engine is necessary, that is, the engines installed on airplanes, trucks, tractors, etc.

We know from physics how a steam engine operates: the steam produced by burning fuel in a steam boiler enters the steam cylinder of the engine, pushes a piston, and forces it to move in the cylinder. A special device is connected with the moving piston and transmits motion, for instance, to the wheels of a locomotive. An internal combustion engine also has a cylinder, in which a piston moves, but it operates as a result of the combustion of fuel inside the cylinder itself. For this reason the engine is termed an internal combustion engine. Figure 16 schematically shows a cylinder and the separate stages of its operation.

At the first stroke of the piston I (a), from left to right, the working mixture, i.e., the mixture of fuel with the amount of air necessary for its combustion, is drawn in through the admission valve. At the conclusion of the suction of the mixture, the admission valve is closed, and the piston commences its second stroke (II). By the end of the second stroke of the piston the mixture has been compressed and reduced several times in volume. Under compression the temperature of the mixture rises to 400-450 degrees C. When the compression of the mixture has been completed, an electric sparkplug placed in the body of the cylinder gives a spark, and the mixture ignites. Gases are formed on combustion of

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the mixture and create pressure in the cylinder.

Let us assume that the pressure of the mixture after compression is 8.5 atmospheres, while the pressure of the gases after ignition may reach 40 atmospheres and above. These gases by their pressure force the piston to move from left to right (stroke III). This is the working stroke of the piston. Both valves are closed during this stroke. At the end of the working stroke the exhaust b is opened, the gases of the burned mixture are forcibly expelled from the valve, and the piston moves from right to left (IV). This motion is called the exhaust stroke.

As will be clear from this description, only one of the four piston strokes is a power stroke, while the other three are preparatory. These three strokes are performed by inertia, and are due to the working stroke. To make the operation of the engine smooth, 2, 4, or more cylinders are used in it, from which motion is transmitted to a common crankshaft.

Under normal conditions the working mixture burns slowly, the flame is propagated at a velocity of 20-30 meters second, and the motion of the piston is smooth. In some cases the combustion of the mixture takes on the character of an explosion, the flame is propagated at a velocity of 2000-3000 meters/second, and the smoothness of piston travel is disturbed. This phenomenon is called knocking.

Knocking is accompanied by the following phenomena:

(1) sounds of metallic knocking in the cylinders and shaking of the engine;

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(2) incomplete combustion of the fuel, as a result of which black smoke is emitted from time to time by the exhaust;

(3) reduction in engine power;

(4) great overheating of the cylinder walls owing to the increased temperature of the products of combustion.

This may also be accompanied by mechanical damage to the various parts of the engine, burning of the pistons, etc.

Knocking leads to increased fuel consumption, reduction of engine power, and rapid wearing of the engine, or even to its breakdown. Knocking must therefore be avoided by all possible methods.

The occurrence of knocking depends mainly on the chemical composition of the fuel. Under certain conditions hydrocarbons become unstable, readily subject to oxidation by the oxygen of the air, and thus form substances that can burn considerably faster than hydrocarbons themselves. Normal paraffin hydrocarbons are the most unstable in this respect and knock the most. The isoparaffin, aromatic and naphthene hydrocarbons, on the other hand, all have high antiknock value.

The antiknock value of the various classes of hydrocarbons, and of fuels in general, are determined on a standard laboratory single-cylinder Vokesch-type test engine, which has an adjustable compression ratio, i.e., the volume B (Figure 17) may be varied at will.

The determination of the antiknock properties of a fuel by this method is based on comparison of the work performed by the

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fuel in the engine with that performed by a definite mixture (standard) in the position of the cylinder at which knocking, as defined by a pinging sound, commences. We take two pure hydrocarbons as standards for the preparation of mixtures with varying antiknock values. One of them, normal heptane, has low antiknock properties, while those of the other, isooctane, are excellent. The antiknock value of a fuel is evaluated by a conventional value called the octane number (from the word octane). The octane number of isooctane is taken as 100, and that of normal heptane as 0. If the fuel being tested knocks in the engine as heptane does, this means that it is a very poor fuel, and its octane number is 0. However, if it behaves like isooctane in the test engine, it is an excellent fuel, with an octane number of 100. Finally, if it behaves like a mixture of 70 percent isooctane and 30 percent normal heptane, this means that its octane number is 70. In determining the octane number of a fuel being rated, a mixture of ixooctane and normal heptane is selected that will knock in the engine in precisely the same way as the fuel to be rated.

Consequently, the octane number of a motor fuel is the percentage by volume of isooctane contained in a mixture of isooctane and normal heptane that will knock in the engine in exactly the same way as the fuel being rated.

We present the octane numbers of a few hydrocarbons.

The saturated hydrocarbons (gaseous hydrocarbons), methane, ethane and propane, have high octane numbers -- 125. Normal butane has an octane number of 91; isobutane, 99; normal pentane, 64;

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isopentane, 83; normal octane, 0; isooctane, 100.

The olefin hydrocarbons have somewhat higher octane numbers than the normal paraffin hydrocarbons: one of the normal hexanes has an octane number of 44, and a second has 100, while that of normal hexane is 23.

Naphthene hydrocarbons likewise have high octane numbers: methylcyclopentane is 70; ethylcyclopentane, 58; cyclohexane, 77; methylcyclohexane, 74, etc.

The aromatic hydrocarbons have the highest octane numbers of all: benzol is over 100; toluol, 110; ethylbenzol and xylol, 112.

Thus the normal paraffin hydrocarbons have the lowest octane numbers, followed by the naphthene, olefin and aromatic hydrocarbons.

14. The Classification of Crudes

The crudes from different deposits vary very greatly in composition and in their physical and chemical properties. For this reason the terms Baku, Grozniy and Emba crudes are meaningless in themselves.

Certain classifications have been worked out in the course of the development of the petroleum industry. The oldest and simplest of these classifications was that adopted by us in Russia: crudes were divided by the commercial criterion into kerosene crudes and kerosene-oil crudes, the former producing kerosene and fuel oil, and the latter kerosene and lubricating oils.

Crudes were subsequently divided into three classes:

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(1) paraffin base crudes with a specific gravity of 0.800-0.810, rich in gasoline and kerosene and containing considerable amounts of paraffin;

(2) asphalt base crudes with a specific gravity of 0.910-0.970, heavy, dark crudes containing only small amounts of bright petroleum products and large amounts of asphaltic-tarry substances;

(3) mixed-base crudes, intermediate between the first two classes.

Soviet crudes could not be accurately characterized by this classification, and therefore what is known as the scientific classification was adopted in 1931 by the USSR. According to it, crudes were divided into three groups.

(1) Methane crudes rich in paraffin hydrocarbons; only the Zykh crude (Baku) and that of South Iskine (Emba) belong to this group.

(2) Naphthendic crudes containing not less than 60 percent naphthene hydrocarbons. Crudes of this type are the best raw materials for producing lubricating oils. Only the crude of Dossor (Emba) belongs to this group.

(3) Purely aromatic crudes are hardly ever encountered in nature. Only the crude of the Verkhne-Chusovskiye Gorodki belongs to this group.

These three groups do not include all the crudes, and therefore the scientific classification distinguishes three other groups of intermediate crudes:

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(1) Methane-naphthenic crudes,

(2) Naphthene-aromatic crudes,

(3) Methane-naphthene-aromatic crudes.

The Surakhan and Groznly paraffin crudes may be classified in the methane-naphthenic group.

All our heavy resinous crudes belong to the naphthene-aromatic group, including Binagad crude, heavy Maykop crude, Kirmak crude, etc.

Light Maykop crude, light Bibi-Eybat crude, and Balakhan crude belong to the methane-naphthene-aromatic crudes.

In 1941 the new standard classification GOST 912-41 was approved. This classifies all crudes according to their content of sulfur, resin and paraffin, and according to the quality of the gasolines, kerosenes and oils produced from them.

According to this classification, Baku oil-crude is termed low-sulfur, resinous, low-paraffin, yielding high-octane aviation gasoline, medium-octane kerosene, medium-index oil. Ishimbay crude is sulfurous, high-resin, high-paraffin, yielding low-octane aviation gasoline, low-octane kerosene and low-index oil.

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CHAPTER FOUR

THE QUALITY OF THE PRINCIPAL FORMS OF PETROLEUM PRODUCTS

15. Forms of Petroleum Products

The petroleum industry puts out a large number of different petroleum products, with more than a hundred different items. This great diversity of petroleum products is explained by their very broad field of application. They are used as different kinds of fuel and lubricants, and are necessary for all branches of the national economy, as building materials, reagents, solvents, illuminants, etc. There is not a single engine, not a single mechanism or machine tool on which they are not used in one form or another.

There is no generally accepted classification of petroleum products. According to their application and purpose these products are divided into a number of groups, the principal ones of which are presented below.

1. Fuel

The majority of all petroleum products (60-70 percent) are used as fuels of various kinds. Fuel in turn may be divided into the following forms:

(1) Gasoline: aviation, motor and tractor (ligroin and kerosene);

(2) Diesel fuel: diesel oil, gas oil, solar oil, motor oil;

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(1) Boiler fuel: boiler mazut, marine mazut, natural gas and manufactured gas.

2. Lubricants

The second great and important group of petroleum products are the lubricants. They may be divided into the following forms:

(1) Lubricating oils for internal-combustion engines -- aviation oils, auto and tractor oils (avtoly), diesel oils and engine oils; for lubricating the cylinders of steam engines (cylinder, vapor, viscosines); for marine engines;

(2) Lubricant mazuts and semi-tars for lubricating railway car axles and journal boxes;

(3) Special lubricating oils: turbine oils, compressor oils;

(4) Industrial oils: vaseline, separator, velocite, spindle and machine oils;

(5) High-consistency oils: solid oil, constalin, special greases.

3. Special-purpose products

(1) Special-purpose oils: transformer oil, white oils (for medicinal and perfumery use), solar oil P (absorptive);

(2) Lubricant-cooling liquids: emulsol, sulfofresol;

(3) Protective anticorrosive lubricants: for firearms, cannon, ammunition, etc;

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(4) Building material: bitumen, Kubrax asphalt;

(5) Solid and semi-solid waxy petroleum products: vaseline, ceresine, paraffin, ozocerite;

(6) Reagents and materials: acidol, napthenic soap, contact;

(7) Illuminant oils and solvents;

(8) Miscellaneous: green soap, gas lampblack, coke, naphthalene.

Let us consider the properties of some of the most widely distributed petroleum products.

16. Aviation and Motor Gasoline

Gasoline is used in internal-combustion engines of the so-called carburetor type. The essential feature of this type of engine is the formation of the working mixture in the carburetor (mixing device) by atomizing the fuel and mixing its vapor with air. After being compressed in the cylinder, the working mixture is ignited by electric sparks.

Gasolines are used in military and civil aviation, for automobiles, motorcycles, tanks, motorboats, etc.

Gasolines must meet a number of requirements.

1. They must evaporate readily, to assure easy starting of the engine, its rapid warming up after starting, and passage from one gear to another without stalling. At the same time, however, excessive volatility of the fuel is harmful, since it leads to great losses in storage and transportation, increases the fire

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hazard and produces vapor lock in the fuel-feed system of the engine. Inadequate volatility of the fuel, on the other hand, is also inadmissible, since in this case it would contain hydrocarbons hard to evaporate that would dilute the lubricating oil fed into the cylinder to lubricate the wearing parts; the lubricant is washed away, thus leading to rapid wear of the frictional parts.

The volatility of a fuel is determined by its fractional composition (Engler distillation). To evaluate the volatility of an aviation fuel, the temperatures at which 10 percent, 50 percent and 90 percent of a sample pass over in an Engler still are taken. The 10 percent fraction is termed the starting fraction and must distill between 50 and 88 degrees C. The upper limit, 88 degrees C, would indicate the gasoline has poor starting qualities, while the lower limit would indicate excessive volatility.

The 50 percent distillation temperature of a fuel determines the duration of the warming-up period after starting the engine. This temperature must not exceed 112 degrees C. The 90 percent distillation temperature characterizes the engine pickup, i.e., its ability to pass rapidly from a small number of revolutions to a large number and vice versa. The 90 percent distillation point of a fuel must not exceed 150 degrees C, since, if it does dilution of the lubricant will occur. 180 degrees C is considered the end point for aviation gasoline and corresponds to a total recovery of 97.5 percent.

The volatility of aviation fuel is also evaluated by determining its vapor tension in a special laboratory apparatus

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(Figure 18). Its vapor pressure must not exceed 350 millimeters. Data on the fractional composition of motor gasolines is given in Table 6.

2. Gasoline must have high antiknock value, which is the basic and decisive characteristic of aviation gasoline. We have already stated that in some cases, when fuel is compressed in the cylinder, detonation occurs. The worse the antiknock properties of a fuel are, the smaller the volume into which it can be compressed. Yet the compression of the mixture before its ignition plays a very great role: the more strongly it is compressed, the better will be the utilization of the fuel.

We shall explain what is meant by the term compression ratio. At the moment when the suction stroke ends, the fuel mixture in the cylinder occupies volume A (see Figure 17). At the end of compression it occupies volume B. The ratio between volume A and volume B is the compression ratio of the engine in question.

Obviously the compression ratio is constant for any given engine, since cylinder volume and piston stroke are both constant.

The influence of the compression ratio of an engine on its power may be judged from the same Figure 17. An engine with a compression ratio of 5 : 1 (in which the volume of the mixture is reduced 5 times on compression) develops 114 horsepower on a consumption of 240 grams of fuel per horsepower-hour. In an hour this motor thus consumes 27.4 kilograms of gasoline. The consumption of fuel in an engine with a compression ratio of 7 : 1 is reduced

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to 200 grams per horsepower-hour, while the power developed by the engine reaches 135 horsepower. This means that the engine uses 27 kilograms of gasoline an hour. Thus, an engine with a compression ratio of 5 : 1 will consume 27.4 kilograms/hour of gasoline to develop 114 horsepower while one with a 7 : 1 compression ratio will use only 27 kilograms of gasoline an hour to develop 135 horsepower.

Why not increase the compression ratio on all engines, then?

The point is that the tendency to increase engine compression ratios is limited by the antiknock rating of gasoline: the higher the engine compression ratio is, the higher must be the antiknock rating of the fuel used. An engine cannot operate on gasoline of a lower octane number than is required by that engine. However, if an engine with a low compression ratio is forced to operate on high-octane gasoline its power will not increase, the consumption of fuel per horsepower hour will not be reduced, and no advantage will be gained from the use of this gasoline. The proper grade of fuel should therefore be used on every engine.

Aviation gasoline is branded by its octane number. Thus, for instance, an aviation gasoline with an octane number of 70 is designated, aviation gasoline B-70. In the USSR aviation gasolines are put out with octane numbers of 70, 74 and 78 (see Table 6).

We have already spoken above of the influence of chemical

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structure on the octane number of a fuel. The higher the content of isoparaffins and aromatic hydrocarbons in a fuel, the higher its octane number. The fractional composition of gasoline also affects its octane number: the lighter the gasoline the higher its octane number.

Aviation gasolines are always used with the addition of substances to increase the octane number, which are called anti-knock agents. Tetraethyl lead (TEL) is generally used for this purpose.

TEL is a heavy colorless liquid with a specific gravity of 1.66, boiling (and decomposing) at 200 degrees C, with a rather pleasant odor. It is a powerful poison, and leads on careless handling to serious nervous disorders and even to death. TEL itself is not directly added to gasoline, but instead what is termed ethyl or lead fluid, composed of 54.6 percent TEL, 36.4 percent ethyl bromide, 9 percent monochlornaphthelene and 0.01 percent sudan dye.

The addition of ethyl fluid to aviation gasoline has various effects on the octane number, the increase in which depends on the character of the gasoline. The degree of increase in the octane number of aviation gasoline on addition of ethyl fluid is called its lead susceptibility. The lead susceptibility of cracked gasolines is low (Table 5), and that of straight-run gasolines is high. Addition of 3 cc of ethyl fluid increases the octane number of carcked gasoline by 10 points, that of Grozniy aviation gasolines by 21-23 points, and that of Baku aviation gasolines by 19 points.

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Table 5

OCTANE NUMBER

Amount of ethyl fluid cc	Baku aviation gasoline	Grosniy aviation gasoline	Cracked aviation gasoline
0.0	70	60	69
0.5	76	69	74
1.0	81	73	76
1.5	84	77	78
2.0	87	79	79
3.0	89	81-83	79

As may be seen from the table, the first 0.5 cc, 1.0 cc and 1.5 cc of ethyl fluid added produce the greatest effect. Further addition of ethyl fluid increases the octane number of gasolines only insignificantly. After the addition of 4 cc of ethyl fluid, no further increase in the octane number occurs, and the use of the gasoline also becomes hazardous. The addition of ethyl fluid is therefore limited to 3-4 cc per kilogram of gasoline.

3. Gasolines must posses high stability under operating conditions and in storage, as well as anticorrosive properties. They must be chemically stable. This means that under conditions of storage and operation they must not acidify, gum or change their fractional composition. The stability of aviation gasoline

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is guaranteed by the norm for actual gum, which must not exceed 2 milligrams per 100 cc. Actual gum is determined in the laboratory by passing a current of air through the gasoline, heated to 100 degrees C in a glass cup.

The residue in the cup after evaporation of the gasoline should not exceed 2 milligrams. Cracked gasoline contains up to 30 percent unsaturated hydrocarbons, which are unstable and resinify. Such gasoline cannot be stored for long periods, and therefore the guarantee period for storage of cracked gasoline has been set at 6 months.

The chemical stability of cracked gasoline is determined by what is known as its induction period. A flask with 100 cc of the gasoline is placed in a metal bomb and subjected to a temperature of 100 degrees C in an atmosphere of oxygen under a pressure of 7 atmospheres. The gasoline is heated until it begins to oxidize, when the manometer will register a fall in the pressure inside the bomb. The gum formed in the gasoline during this time represents the potential gum. Cracked gasoline is considered standard if the induction period is 240 minutes, i.3., if the manometer reading remains constant for 240 minutes.

Table 6

The Quality of Gasolines

	B-78	B-74	B-70	KB-70,	Motor gasoline,
	GOST 1012-44			GOST 2069-43	GOST 2084-46
	[1]			[2]	[3]
Octane number of gasoline in pure state	78	74	70	70	--

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Table 6 (cont'd)

	[1]			[2]	[3]
Same on addition of not more than 4 cc of ethyl fluid	95	92	89	--	--
Initial boiling point, degrees C	40-75	40-75	40-75	minimum 35	--
10%, degrees C, max.	86	88	88	88	85
50%, degrees C, max.	112	112	115	118	150
90%, degrees C, max.	150	150	150	150	210
End point, degrees C	180	180	180	175	225
Vapor pressure, mm of mercury, max	330	330	330	375	375
Acidity, mg. KOH per 100 cc	1.2	1.2	1.5	1.8	--
Freezing point, degrees C, max	-60	-60	-60	--	--
Induction period, minutes	--	--	--	800	240
Actual gum content, mg. per 100 cc	2	2	3	6	10
Sulfur content, max, %	0.05	0.05	0.05	0.05	0.15
Mechanical admixtures: water, water-soluble acids and alkalis	ABSENT				
Color and transparency	COLORLESS AND TRANSPARENT				
Copper-strip test	NO CORROSION				

Gasolines must not have corrosive properties, i.e., the ability to attack metal. For this reason all sulfur compounds and other harmful substances are eliminated from gasolines.

Gasolines must also pass the copper-strip test, as well as the so-called doctor test.

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Among the other qualities required of aviation gasoline, the following must be noted: maximum freezing point -60 degrees C, absence of water-soluble acids and alkalis, absence of mechanical admixtures and water, colorlessness and transparency. Table 6 gives the principal qualities of aviation gasoline.

The specifications for cracked motor gasoline are less severe. They do not require the determination of color, transparency nor freezing point, and no octane number is prescribed. The qualities of motor gasoline are given in the same table.

To improve the quality of cracked gasoline, antioxidants are added to it to retard the formation of gum. Table 6 gives the specifications for gasoline KB-70, which is used in tanks (and therefore termed tank gasoline).

17. Tractor Fuel

At the present time kerosene is the principal tractor fuel. Ligroin and diesel fuel are also used with some types of tractors.

Ligroin is a product of straight-run distillation. It must also have a high octane number, not less than 54. It is considerably heavier in its fractional composition than the gasolines: not less than 12 percent distills up to 150 degrees C, not less than 90 percent up to 200 degrees C, and the maximum end-point is 230 degrees C. Since ligroin (like kerosene) lacks a starting fraction, the motor of a tractor is started up on motor gasoline. Ligroin must be free from water-soluble acids and alkalis, sediment and water; it must pass the copper-strip test and have an acidity no higher than 4 (in milligram KOH).

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Kerosene is still heavier than ligroin. The most important characteristic of tractor kerosene is its octane number, which must not be less than 40. Our plants put out two grades, I and II. 10 percent of grade I distills below 200 degrees C, and 20 percent of grade II. The end point of grade I is 300 degrees C, and that of grade II is 315 degrees C. 80 percent of both grades pass over at 270 degrees C.

Besides the straight-run kerosenes a cracked tractor kerosene is also put out, with an end point of 275 degrees C and an octane number of 30.

Illuminant kerosene is distinguished from tractor kerosene by being more highly refined; it is also heavier, with initial boiling point at 220 degrees C and an end point of 315-325 degrees C. In all of its other properties it closely resembles tractor kerosene. No octane number is prescribed for it but this is always considerably lower than 40.

18. Boiler Fuel

In comparison to solid fuel, oil fuel possesses immense advantages, of which the principal ones are:

(1) simplicity and ease of regulating oil furnaces, which require only a third to a quarter of the attendant personnel that coal furnaces do;

(2) complete combustion of the fuel and absence of ash;

(3) cleanliness in the buildings as a result of the absence

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of ash and dust;

(4) considerably less storage space for fuel is needed;

(5) high heat value, up to 10,000 Cal/kg (1½-2 times that of coal).

Boiler fuel is burned in special devices called burners. (Figure 19). Burners consist of two tubes, one within the other. Fuel is fed through the inner tube, and steam through the outer, thus atomizing the fuel and producing a flame-jet several meters long.

Boiler mazuts are classified according to their viscosity at E_{50} into the following marks: mazut 10, 20, 40 and 80. These figures indicate the viscosity at 50 degrees C.

The flash point of mazut ranges from 65 to 110 degrees C (by Brenken tester). The higher the viscosity of mazut, the higher its flash point. The freezing point also increases from + 5 degrees C in the low-viscosity mazuts to + 25 degrees C in high-viscosity mazuts.

The ash-content of mazut is low and amounts to 0.3 percent for all types of this fuel. The sulfur content is limited by the standard to 0.5 percent, but the crudes of the Eastern regions give mazuts with sulfur over 3-4 percent. In some cases even such mazut must be used.

Cracked mazut is a high-viscosity, very resinous fuel containing particles of coke. Its use, especially in winter, involves

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a number of inconveniences: congelation in the supply pipes, stopping up the burners with coke, etc.

19. Lubricating Oils for Internal-Combustion Engines

Friction and Lubrication

The surface of any mechanism, no matter how well polished it is, still remains rough. Microscopic examination of a smooth surface reveals projections and hollows invisible to the naked eye. If two such surfaces move relative to each other, they produce what is termed dry friction, causing wear on the bearing surfaces, overheating and expenditure of considerable energy in overcoming friction.

To eliminate dry friction between bearing surfaces, a film of lubricating oil is applied to them. Fluid friction then arises, and the wearing surfaces make no contact whatever with each other; consequently, no wear will result. Wearing surfaces may operate under entirely different conditions: at high, low or room temperatures; at high or low pressures; in the presence of water vapor, reagents or air; at high or low engine speeds.

Each set of operating conditions for a mechanism requires an oil of the appropriate qualities. For instance the same oil cannot be used to lubricate a welding machine and a locomotive, or a motor operating in the Far North and one operating in tropics, etc. A low-viscosity oil is needed to lubricate rapidly rotating spindles, while a steam pump operating with superheated water vapor at a temperature of up to 300 degrees C needs a very viscous oil with a high flash point. Our industry puts over 50

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different kinds of oil on the market, which are used for lubricating various mechanisms operating under the most diversified conditions.

The most extensive and critically important group of lubricating oils is made up of oils for internal-combustion engines. They are used for lubricating the cylinders of airplane, automobile, tractor and diesel engines. Motor oils, i.e., oils for lubricating the cylinders of automobiles and tractors, are produced in the greatest quantities.

Viscosity is a very important quality of oils. If a motor is to start easily in cold weather, and minimum friction in the engine parts is to be assured, the viscosity of the oil cannot be high. On the other hand, if the viscosity of an oil is low, it can easily be forced away from the bearing surfaces. The variation of viscosity with temperature is no less important. Consequently, the viscosity of an oil should be so chosen as not to impede starting of the engine in cold weather, and yet be sufficient under operating conditions to lubricate even the hottest parts of the engine. As we have already learned, this demand is satisfied by an oil with a high index of viscosity.

Motor oils are put out in four marks: 4, 6, 10 and 18. These figures indicate the mean viscosity of the oils at 50 degrees C. The freezing point of motor oils varies from -- 30 to 0 degrees C, and their flash point from 185 to 215 degrees C. The flash point and the freezing point both increase with increasing viscosity.

The color of motor oils must be from 2 to 3 millimeter

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(determined by colorimeter).

Motor oils are tested for coke formation, i.e., for the tendency of oils to form carbon deposits under operating conditions.

Coke formation is determined as follows. (Figure 20). A certain amount of the oil is heated by a strong flame in a metal crucible without the access of air. All the volatile components are thereby eliminated, leaving only coke in the crucible. The more coke remains there, the poorer the quality of the oil. For motor oils coke formation from 0.3 percent (motor oil 6) to 0.7 percent (motor oil 18) is allowable.

Different marks of motor oils are used on trucks and tractors at different seasons. In summer the most viscous oil, motor oil 18, is used on all tractors except diesels. In winter, motor oil 10 is used on them. In summer, motor oil 10 is used on automobiles, and motor oil 4 or 6 in winter.

Aviation oils must have low freezing points to assure easy starting of the engine in winter (from -- 11 to -- 30 degrees C.), high flash points (200 to 300 degrees C), and must be highly refined, assuring stability of the oil on long standing and under operating conditions.

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CHAPTER FIVE

THE BASIC PROCESSES AND APPARATUS OF PETROLEUM REFINING

Characteristics of the basic processes and apparatuses of petroleum refining.

The following operations must be performed to obtain petroleum products from crude oil:

(a) The crude must be transported from the reservoir to the refinery.

(b) It is then preheated in heat exchangers, using the heat of mazuts and distillates leaving the installation.

(c) The crude is next heated in heating apparatus.

(d) It is evaporated and separated into various fractions in rectifying columns.

(e) The vapors are condensed and the hot liquids cooled.

(f) The products of distillation are transported into containers.

The products of distillation are called distillates, and are not yet commercial products. The distillates need to be treated to free them from the harmful substances they contain.

In the treating plants (a) the distillates are treated with reagents, and (b) the products of treatment are separated from the waste.

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Various processes -- chemical, adsorptive, solvent -- may be employed in treating the distillates with reagents, according to the methods of treatment. These processes will be discussed in Chapter X.

1. STORAGE AND PUMPING OF CRUDES AND PETROLEUM PRODUCTS

20. Storage of Petroleum Products and Accounting for them

Cylindrical metal containers called tank are used for storing crude oil and petroleum products (Figure 155).

Crude is stored in large tanks (of capacity up to 12,000 cubic meters). Petroleum products are stored in smaller reservoirs holding up to 1,000-2,000 cubic meters. Small reservoirs holding 25-200 cubic meters used to receive products from the refinery are called receivers. Each reservoir is provided with receiving, discharging and level-lowering lines, adapted for the breathing of the reservoir, for the extinguishing fire, and for gauging the petroleum products, and is also provided with ladders, platforms and manholes.

The receiving line may be located 1 to 2 meters above the bottom of the tank. The discharging line is usually 30 to 40 centimeters above the bottom of the tank, so as not to take water and sediment.

The level of the tanks may be measured by various methods: (a) by float devices, (b) by metering cocks, (c) by a gauge.

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The gauge is the most widely used method of measurement. A metal gauge is lowered through a manhole in the roof of the tank and the level of the product measured. At the same time its specific gravity and temperature is also measured.

Assume that the measurement showed a depth of 310 centimeters, that the temperature of the product is 50 degrees C, and that its specific gravity at this temperature -- or, as it is called, its submersion -- is 0.820. We calculate the specific gravity by the aid of Table 3, and find it to be 0.841 at 20 degrees C.

Assume that 1 linear centimeter of the reservoir contains 0.357 tons of the product. We determine the amount of the product in the reservoir:

$$310 \cdot 0.357 \cdot 0.841 = 105 \text{ tons.}$$

On storage of light petroleum products in reservoirs, large evaporation losses are possible. Such losses are particularly large during the storage of gasoline. When the outside temperature rises, the gasoline becomes heated, and gasoline vapors are formed, which escape through the manholes and breather valves on the roof. When the outside temperature falls, on the other hand, the gasoline vapor condenses, thus causing rarefaction inside the tank and sucking air into it. Thus the escape of gasoline vapor from the tank alternates with the suction of air into it. As they say, breathing of the tank takes place and leads to the loss of petroleum products.

The following methods are used to combat losses of petroleum

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products: (a) spraying the tanks with water, (b) installation of breather valves, (c) insulation of the tanks (to maintain constant temperatures), (d) use of a floating roof that falls and rises with the product, (e) painting the tanks in light colors. Practice has shown that twice as much gasoline is lost when the tanks are painted black as when aluminum paint is used.

21. Pumps

The piston pump and the principle on which it operates

The steam piston pump is the most widely used pump in oil refineries.

The steam piston pump (Figure 21 at the top of the scheme in the center section) has two cylinders: the hot or steam cylinder A, and the cold or product cylinder B. The cold cylinder has the piston C and two rows of valves -- the receiving or suction valves K and the discharge valves M. The hot cylinder is provided with a piston, a valve and valve chest. The pistons of the cold and hot cylinders are connected by a common piston rod. The valve chest is provided with two passages to deliver steam to the cylinder: one of them introduces steam in front of the piston (the left side of the cylinder) and the other in back of the piston (the right side of the cylinder). In passing into the left-hand portion of the piston, steam under pressure pushes the piston to the right until it reaches the extreme right position. At this moment the valve, moving left, closes the left aperture in the slide box, and opens the right aperture. The steam then enters

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the right-hand portion of the cylinder and pushes the piston to the left. At the same time the exhaust steam from the left-hand portion of the cylinder is ejected through a special vent. When the piston reaches the extreme left position, the left-hand aperture in the slide chest is opened (the right one being already closed), and the piston therefore moves to the right again, and so on. Thus, the piston moves uninterruptedly right and left, forcing the piston in the product cylinder of the pump to move in the same way.

The product part of the pump operates as follows: when the piston moves to the right, rarefaction occurs in the left hand portion of the cylinder, the admission valve K is opened and sucks the liquid into the cylinder; when the piston moves to the left, pressure is produced, under the influence of which the suction valve is closed, the discharge valve M is opened and the liquid is forced out into the discharge line. Since there are admission and discharge valves on both sides of the piston, both sides of the cylinder operate alternately to suck in the liquid and force it out, i.e., during the time when the left side is sucking in the liquid, the right side is expelling it. Consequently, both strokes of the pump are working strokes.

Piston pumps may also be power driven, in which case the piston is actuated by an electric motor.

The cold part of the pump may be either single-cylinder or two-cylinder. The size of the cylinder and piston vary in accordance with the required capacity of the pump.

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The centrifugal pump and the principle on which it operates

The centrifugal pump consists of the body B and the working wheel A (Figure 21, bottom). The working wheel consists of two parallel bronze or cast-iron plates, between which there are a row of oblique baffle-vanes. It is attached to a shaft passing through the center of the body and resting on two bearings.

When the pump operates, its body and receiving line E are so filled with liquid as to leave no air in them. As the wheel rotates rapidly, the vanes force the liquid upwards into the discharge line M, while beneath, in the receiving line, a vacuum is formed, as a result of which the following portion of the liquid also reaches the working wheel.

Centrifugal pumps may be single-stage (with one working wheel), and multi-stage (with several working wheels on a single shaft O). Single-stage pumps are low-pressure, while multi-stage pumps are high-pressure (over 5 atmospheres).

II. Heat-exchangers, condensers and coolers

The Significance and Characteristics of Heat-Exchange Apparatus in Oil Refineries.

The principal sources of heat in oil refineries are pipe stills and steam boilers.

Either unit makes it possible to utilize the waste heat of vapors and hot liquids which must be cooled before they enter the tanks.

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The process of heat exchange takes place in heat-exchange equipment which is of varied design and is classified according to purpose into:

(1) heat exchangers or heat regenerators, which are devices designed to heat raw materials by cooling emerging hot liquids and vapors; in the former case they are termed liquid heat exchangers, while in the latter case they are termed vapor heat exchangers.

(2) Condensers -- apparatus for condensing vapors in most cases with the aid of water.

(3) Coolers -- apparatus for cooling hot liquids by means of water.

According to their design, heat-exchange apparatus is classified into:

(1) coil,

(2) pipe,

(3) mixture condensers.

Coil-type heat-exchange apparatuses are either sprayed-coil type or coil-in-lot type. They are mainly used as condensers and coolers, and less frequently as heat exchangers.

Pipe-type heat-exchange apparatus are pipe-inpipe type, single-pass, or multi-pass. They are used as heat regenerators, and less frequently as coolers and condensers.

Mixture condensers are used to condense vapors by direct

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mixture with water in the apparatus.

Heat Exchangers or Heat Regenerators

The quantity of heat utilized in heat exchangers depends on a number of conditions, of which the principal ones are as follows:

(1) The temperatures of the hot and cold currents -- the greater the temperature difference, the greater the quantity of heat that will be transferred from the hot current to the cold one;

(2) the speed with which both currents move -- the faster they move, the better will the heat exchange be;

(3) viscosity -- the lower the viscosity of the liquids, the better the heat exchange;

(4) the design of the heat exchanger; under the very same conditions the heat exchanger may utilize a larger amount of heat with a smaller surface of the apparatus, merely on account of modifications in design. This may be achieved by means of counter-current, baffle-plate arrangement, increase in flow velocity, etc.

During the course of technical progress, heat exchangers have replaced each other in the following sequence: coil type, pipe in pipe, single-pass shell-and-tube, multi-pass shell-and-tube.

Coil-type heat exchangers were formerly widely used on account of their uncomplicated design and simplicity of servicing. The coils, consisting of 2 to 3 inch pipes, were placed in a tank

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or in a horizontal preheater with the crude oil. The hot mazut was pumped from the crude-oil preheater through the coil. The heat transfer in such a heat exchanger was inefficient, since the crude was almost motionless while the hot product was pumped through at low velocity. Bulky apparatus therefore had to be used to transfer the necessary amount of heat. At the present time this method of heat exchange is only used in the very oldest installations.

Pipe in pipe heat exchangers consist of apparatus with double pipes; the hot product flows through one of them, while the cold product flows through the other (through the ring-shaped space). The pipes are arranged in offset (Figure 22) for convenience in servicing. 2-inch pipe inside 3-inch, and 3-inch pipe inside 4-inch, are the sizes ordinarily used.

These heat exchangers are bulky and expensive, since they require the use of a large amount of metal in their construction. At the present time this type is only used in cases where the liquids form a considerable amount of deposit on the pipe walls (for instance, of coke when cracking residues, or when heavy paraffin is being passed, or paraffin crudes are being treated, etc). The pipes are easily cleaned of such deposits.

The most widely employed heat exchangers are the very efficient tubular type.

The tubular heat exchanger consists of a steel cylindrical body with a diameter up to 500 millimeters, through which pass a

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bank of seamless steel tubes of 18-25 millimeters diameter (Figure 23). The ends of the tubes are rolled into tube plates welded to the body.

In single-pass heat-exchangers the crude enters the connecting branch 1, passes through all the tubes of the heat exchangers, and is then conducted through the connecting branch 2 into the following heat exchanger. The hot product enters the connecting branch 3 at one end of the heat exchanger and leaves it through the connecting branch 4 at the other end, so that the streams of hot and cold liquid flow countercurrent. One disadvantage of such heat exchangers is the poor heat transfer owing to the low velocity of the streams.

This disadvantage is eliminated in multi-pass heat exchangers by the installation of baffles. As will be seen from Figure 24, the heat exchanger has one baffle in the left-hand distribution chamber, and two of them in the right-hand side. The crude enters through the connecting branch 3, makes one passage through one section of the tubes, then makes a turn (8) and enters the next section of pipes, located higher; in the right-hand distribution chamber the crude makes a turn to the left (7), and finally makes one more turn in the left-hand distribution chamber. As a result the crude, having made four turns in all, leaves through the connecting branch 4. The body of the heat exchanger likewise contains the baffle 2, which forces the hot liquid to make two passages. The mazut enters through pipe 5 and leaves through pipe 6.

This heat exchanger is a four-pass type in the tubular space

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and a two-pass type in the shell space. The Bakinskiy Kabochiy type of heat exchanger that is widely used in the USSR belongs to this type. It has the following technical characteristics:

Useful length of tubes	5930 mm
Number of tubes	112
Inside diameter of tubes	20 mm
Outside diameter of tubes	25 mm
Heating surface	52 square meters
Inside diameter of body	457 mm
Number of passes through bank of tubes	4
Number of passes through shell space	1

Vapor heat-exchangers are designed to utilize the heat of condensation of vapors for heating crude. They are placed on top of the column or beside the evaporator (in the case of cracking plants). In Winkler-Koch system cracking plants, vapor heat exchangers with a shell diameter of 920 millimeters and length of 4.23 meters are installed. Inside this shell there is a bank of 196 tubes 20 millimeters in diameter, with 52 square meters of heating surface. This heat exchanger is four-pass. Two such heat exchangers in Winkler-Koch plants heat mazut from 20 to 220-250 degrees C.

With regard to the choice of current direction let us consider a few cases of the most advantageous distribution of the currents between the tube and shell spaces.

1. Products occupying a large volume (gases and vapors) are more easily passed through the shell space, since its total area is greater than the total area of the tubes.

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2. The most viscous liquids are more easily passed through the shell space.

3. Liquids are passed through the tubes in cases where there is a deposit of scale, coke, sediment or other substances, since it is easier to clean out such deposits from the tubes than from the shell space.

4. If the liquid possesses corrosive properties, it must also be passed through the tubes. In this case the tubes are made of a special alloy, or, if they are of ordinary steel, they are frequently replaced. This is cheaper than replacing the whole shell if the liquid passes through the shell space.

5. Where the stream of liquid must be fed under high pressure it should be passed through the tubes.

23. Condensers

Condensers are designed to condense the vapors of distillates. The following designs are known:

(1) tubular condensers,

(2) submerged condensers (coil-type and heater type),

(3) mixing condensers.

Tubular condensers are mainly designed to utilize the heat. One of these heat regenerators, the vapor heat exchanger, is described in Section 22. This type of equipment is also known as partial condensers, since they do not condense all the vapors, but only their heavier portions. In cases where all the vapors

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are to be condensed, water is passed through the condenser tubes and the vapors through the intertube space.

Tubular condensers occupy little space, require relatively small amounts of metal, and are cheaper than the bulky submerged condensers. In spite of these advantages, the basic disadvantage of fire hazard that attaches to them has prevented them from being very widely used. The supply of water in tubular condensers is negligible, and if the feed of water is interrupted, the vapors are unable to condense, pass into the receiving compartment and create the danger of fire there. The condenser tubes are also frequently out of commission on account of corrusion by water.

Submerged condensers (Figure 25) consist of metal boxes containing continuous coils or headers with separate coils.

In the coil-in-box condenser, the vapors pass in a single continuous stream through the entire coil. In the header condenser the vapors pass through a tube of large diameter, the header, and then branch off into a series of parallel streams through separate coils. All of these streams are then joined again into a single stream in another header. The header condenser is considerably more convenient, since if a coil goes out of commission it can be cut out of service with only a short halt in plant operation.

The coils of submerged condensers are usually made of 3 to 4 inch cast-iron pipe, which serve for protracted periods without replacement. Water is supplied to the condensers in an upward stream from the bottom. The large supply of water in the box

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allows shutting down the plant without risk of damage, in case the water feed should be interrupted. Moreover the submerged condenser is preferable to the tubular condenser in cases where the water supplied to the equipment is muddy or contains large amounts of salts corrosive to metal. Deposits of salts, mud or rust are cleaned out of submerged condensers with considerably greater ease than from the tubular type.

Mixture condensers (Figure 26). In mixture condensers the heat from the vapors of petroleum products is transferred to water by direct contact between the vapor and liquid phases. The greater the contact surface between the two phases, the better the heat exchange.

A greater contact surface between vapor and liquid is obtained by two methods: (1) by finely atomizing the water, which is delivered in the form of a spray, and (2) by installing a checkered brickwork of Rashig rings, or broken brick, etc.

In a mixture condenser the vapors and gases rise upward from the bottom, countercurrent to water supplied from the top. At the bottom of the condenser two layers of liquid are formed: the water and the distillate. The upper layer, the distillate, is pumped out into the reservoir, while the lower layer, the water, is drained off into the sewer.

Mixing condensers are mainly used for condensing light petroleum products, since a mixture of water with heavy petroleum products is not easily separated into its constituents on account of the slight difference in the gravity of these liquids.

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24. Coolers

There are the following designs of coolers: (1) tubular, (2) submerged, (3) spray.

Tubular coolers are constructed like heat exchangers and are termed final coolers. They are mostly used for cooling light distillates.

Submerged coolers (see Figure 25) are constructed the same as submerged condensers. A general view of one of them is shown in Figure 27.

Spray coolers (Figure 28) consist of continuous coils or of collectors with separate coils. The coils are set up in a concrete pit with an outlet to the drain system, and are sprayed with water from overhead channels. The water in flowing downward from above, forms a film on the pipes which efficiently removes heat from the product through the rapid change of water and its partial evaporation. Air is blown through the spray coolers and helps to cool the distillate. The consumption of water by spray coolers is very low, which is very important for plants that do not dispose of adequate quantities of water.

The disadvantages of spray coolers are the strong corrosion of the pipes under the combined action of water and air, as well as the influence of atmospheric conditions.

25. The operation of heat-exchange equipment

The principal task of the attendants at a petroleum distil-

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lation plant is to check the cleanliness of the heat-exchange equipment and at the same time to perform the necessary cleaning, since fouling is the main cause of their poor operation. Deposits of coke on the tube walls lower the transfer of heat from the hot liquid to the cold. For instance the transfer of heat through a tube coated with a layer of coke 2-3 millimeters thick is several times lower than through a clean tube.

The following deposits accumulate in heat-exchange equipment:

Solid deposits: from water -- scale, rust and corrosion products; from the distillate -- coke;

Porous deposits: porous coke, porous scale;

Loose deposits: slime, mud, soot, coke powder; some of these deposits are readily detached by blowing steam through or washing out with water.

The hard and porous deposits can be removed only by mechanical means or by washing out with chemicals; washing out with hot water or blowing steam through will not remove them.

Cleaning is done as follows. The bank of tubes (Figure 29) is removed from the shell and all the deposits in the shell space are scrubbed off and cleaned with scrapers or metal brushes. The inside of each tube is cleaned with long metal rods.

In cleaning submerged condensers and coolers, the water is let out of the box, and the mud and slime accumulated in it is

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taken out; all the deposits are scrubbed off by mechanical means and removed from the box.

Heat-exchange equipment operates at a considerably higher efficiency after cleaning.

III. The working principles of the pipe still

26. Characteristics of the Pipe Still

The simplest apparatus for heating water is the steam boiler, consisting of a horizontal cylinder containing a tube for burning fuel called the fire tube. The heat produced by the combustion of fuel in the jet burners is transmitted to the water through the walls of the fire tube. The water is evaporated and passes from the boiler into the steam main. To keep the water level constant, fresh water is fed into the boiler at the same rate with which it evaporates.

The steam boiler is a bulky and uneconomic device of low productivity. It was replaced by the water-tube boiler.

The water-tube boiler consists of a bundle of seamless tubes placed in the furnace and bathed directly by the flames. Water under pressure passes through the tubes, and is evaporated on leaving the boiler. At the present time these boilers are extensively used.

Petroleum distillation developed along the same lines: crude oil was distilled at first in ordinary steam boilers called stills. Up to the 30's of the present century the shell still was the principal heating apparatus used, in spite of a number of

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inherent disadvantages: bulk, low productivity, lack of economy, fire hazard. Pipe stills were then constructed. There is a great difference between them and water-tube boilers. Water is heated in the latter and excessive heating has no unfavorable effect on water, but in pipe stills crude, mazut and petroleum products are heated, and if these are overheated they may deposit coke which clogs the tubes and puts them out of commission. Therefore, even the very earliest models of pipe stills were not mere copies of the water-tube boiler.

There are a large number of different pipe stills known today. Every pipe still has two chambers: (1) a combustion chamber where the radiant tubes are placed and (2) a convection chamber where the convection tubes are placed.

Figure 30 shows one of the designs most widely employed in the USSR, a radiant-convection type pipe still with a vertical flow of the combustion gases. Its left-hand portion, which is larger, consists of the combustion chamber, while the right-hand portion, filled with tubes, is the convection chamber. The two chambers are separated by a brick partition wall which does not go all the way to the top of the pipe still. The radiation tubes, arranged in two rows on the ceiling of the combustion chamber, are termed the ceiling bank. 1. The tubes in the convection chamber are arranged in three sections, upper, middle and lower, 2, 3 and 5. The coil-pipe 4, for superheating the water vapor, is located between sections 3 and 5.

The cold crude enters the lower section, 9, of the convection chamber, 9, of the convection chamber, passes upwards through all the sections, enters the lower set of radiant ele-

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ments, and finally emerges from their upper set 6. The burners are placed at the bottom of the front wall of the combustion chamber. The combustion gases, in giving up their radiant heat to the radiation tubes, ascend cross over the partition wall, and bathe all the tubes in the convection chamber. Heat is transferred to this chamber by means of convection. After passing all the pipes in the convection chamber, the combustion gases first enter the horizontal flue and then the smokestack.

The principle of countercurrent flow is followed in the pipe still, i.e., the crude oil moves to meet the combustion gases, and in the opposite direction from them.

27. The Conditions for Fuel Combustion

Mazut and gas, both burned in jet burners, are the fuels used for pipe stills.

The combustion of fuel is the chemical reaction of the organic compounds of the fuel with the oxygen of the air. The oxidation reaction is very rapid, developing high temperatures, running up to 1,400 degrees C. The approximate ultimate analysis of mazut is: carbon 86 percent, hydrogen 13 percent, other elements up to 1 percent and more. Mazut from heavy or sulfurous crudes may contain more carbon and sulfur, and less hydrogen.

The combustion of the separate elements of fuel takes place according to the following equations:

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contact with the metallic surfaces of the tubes (to avoid burning them).

In selecting the volume of the combustion chamber we proceed from what is called the rate of heat liberation of the furnace, i.e., the number of large calories liberated per hour, on combustion of fuel, per cubic meter of combustion chamber volume. The rate of heat liberation for pipe stills is taken as 30,000-50,000 Cal/cubic meter hours.

This may be illustrated by the following example.

We know (See Section 9) that the heat value of a fuel may be computed by the (abbreviated) Mendeleyev formula

$$Q = 81C + 246H - 26\,O.$$

We find the heating value of mazut of the ultimate analysis given above:

$$Q = 84 \times 86 + 246 \times 13.0 - 26 \times 0.8 - 9880 \text{ Cal/kg.}$$

2000 kilograms an hour of this kind of fuel are consumed, producing a total amount of heat:

$$9880 \times 2000 = 19,760,000 \text{ Cal/hour.}$$

We assume a rate of heat liberation equal to 40,000 Cal/cubic meter hour. Then the volume of the firebox space will be equal to

$$19,760,000 : 40,000 = 494 \text{ cubic meters.}$$

The linear dimensions of the pipe still are fixed on the basis of the following considerations. The length of the firebox

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from the jet burner to the partition wall must not be less than 4 meters (at minimum flame length). This protects the wall from the impact of the flame, and from destruction. The width of the pipe still is determined in accordance with the length of the screen tubes and may reach 10 meters or more.

In our case let us take a firebox length of 5 meters, and a chamber width of 10 meters. Then the height H of the pipe still will be

$$H = \frac{494}{5 \times 10} = 9.9 \text{ meters.}$$

We have thus computed all the dimensions of the combustion chamber of a pipe still burning 2000 kg/hour of mazut with a heating value of 9880 Cal/kg.

The volume of the combustion chamber usually amounts to 85-90 percent of the total volume of the pipe still, while only 10-15 percent falls to the part of the convection chamber.

Highly productive pipe stills consume much air. To render their operation economical, the air is preheated in recuperators (heat-exchangers for gases) through the cooling of the combustion gases. Fans are used to facilitate the delivery of air to the combustion chambers.

The smokestack is of great importance for the normal operation of a pipe still. It creates a draft, i.e., it draws the combustion gases out of the pipe still. The higher the smokestack, the more powerful the draft and the more efficient is the combustion of the fuel.

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28. Conditions of Heat Transfer in Pipe Stills

The purpose of the pipe still is to heat the crude to the temperature at which the assigned quantity of the light fractions will be evaporated from it on its transfer to the evaporator. This task must be performed with minimum fuel consumption, and in the shortest possible time. As we know, the crude is heated in two chambers of the pipe still, the convection chamber and the combustion chamber. Let us now discuss the conditions of heat transfer in each of these chambers.

The temperature of the gases in the zone of fuel combustion reaches 1,300-1,400 degrees C. Over the partition wall the temperature of the combustion gases falls to 700-850 degrees C, and, finally, when they emerge from the convection chamber, their temperature is 300-400 degrees C.

Heat Transfer in the Combustion Chamber

The radiant energy obtained on the combustion of the fuel is the main source of heat in the combustion chamber. It is transferred to the tubes, the pipe still walls and the combustion gases. The walls and gases in turn transfer some of this heat to the tubes. Thus, the radiation tubes receive up to 80-85 percent of their heat on account of radiation. However, since the radiation tubes at the partition wall are bathed by the combustion gases, they also receive a certain amount of their heat by convection.

The radiant surface of the tubes is increased as much as possible. To this end not only are roof tubes installed, but also

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side wall tubes and even floor tubes (on the furnace bottom). In a pipe still of the type described, radiant tubes make up as much as 50 percent of all tubes. In other furnaces this amount is even greater.

Two rows of tubes are usually installed in the ceiling bank, though sometimes only one. No advantage would be gained by increasing the number of rows of radiant tubes: about 50 percent of the heat received by the first row arrives at the second row in a two-row bank, while in a three-row bank the third row receives only 12-16 percent of the heat received by the first row.

The heat supplied to the radiant tubes should be uniformly distributed over the entire surface of these tubes. The quantity of heat attributable to 1 square meter of tube surface is termed the rate of heat supply of radiant tube surface, and usually ranges from 20,000 to 40,000 Cal/square meter hour. Exceeding this rate of heat transfer to the radiant-tube surface will lead to burning (hot spots).

As we have already pointed out, the temperature of the combustion gases at the partition wall sinks to 700-850 degrees C. In some pipe stills the combustion gases are recirculated to obtain a mild, smooth regime. Such gases are collected from the horizontal flue by means of a fan and are fed again into the combustion chamber, where they mix with the burning combustion gases and reduce their temperature. Sometimes the waste combustion gases are supplied in an amount equal to 100 percent to 300 percent that of the fresh combustion gases. The recirculation of heat reduces the fuel consumption and improves the operation of

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the convection chamber.

Heat Transfer in the Convection Chamber

Heat is transferred in the convection by immediate contact of the hot gases with the tubes.

Heat transfer by convection depends on the velocity of the gases. The greater the quantity of gases passing through the chamber, the more heat they will give up to the tubes. The recirculation of gases therefore encourages the better transfer of heat by convection.

The transfer of heat by convection also depends on the cleanliness of the tubes, since fouling of the tubes impedes heat transfer.

The rate of heat supply to the heating surfaces of the convection tubes ranges from 8,000 to 12,000 Cal/square meter-hour.

There is some heat lost in the still. A certain part of the heat obtained by the combustion of the fuel passes through the cover of the pipe still on account of its thermal conductivity. The better the pipe still is insulated the lower this wasted proportion of the heat will be. In general these losses may amount to 5-12 percent of all heat liberated in the pipe still. The greatest heat loss is in the combustion gases (20-30 percent). Thus, as a rule, after deduction of these losses, the useful utilization of heat amounts to 65-75 percent.

The Heat Balance of the Pipe Still

Let us analyze for illustrative purposes the heat balance

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of a radiation-convection-type pipe still with a vertical flow of gases. Figure 31 shows how the heat obtained by combustion of fuel is distributed (1). 36 percent of the heat (3) is transferred to the radiation tubes from the combustion mirror, and 11.2 percent by radiation from the combustion gases (5). Besides the heat of radiation, the radiant tubes also receive 10.1 percent of the heat by convection (6). The upper convection chamber receives 7.3 percent of the heat (8). The two lower convection sections receive 2.65 percent of the heat (10), while 2.8 percent of the heat is expended in superheating the steam in the superheater (9).

Thus, 77 percent of all the heat is expended usefully. Of this amount the radiant tubes receive 57.3 percent, the convection tubes 16.9 percent, and the steam superheater 2.8 percent.

In the case involved the heat losses amount to 22 percent. This sum is made up of the losses through the combustion gases (11), 14.8 percent and the losses to the surrounding medium, 8.2 percent; of these, 3.6 percent is lost in the lower half of the combustion chamber 2, 3.6 percent in the upper half of the combustion chamber 4, and 1 percent in the convection chamber 7.

29. Types of Pipe Stills

In the USSR, the first pipe still was constructed at Baku in 1925. It was the pile type with a removable combustion chamber and low productivity (Figure 32, 1). The transfer of heat in this and similar pipe stills is mainly accomplished through convection.

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The next step in the development of the pipe still was the construction of one with a removable combustion chamber 2, and then another, a convection-type still, with a small combustion chamber (see Figures 32, 33, 34). These pipe stills operated very inefficiently, with small coefficients of heat transfer and low utilization of useful heat.

When the role of radiation in heat transfer in pipe stills at length became clear, the construction of pipe stills of the radiant-convection-tube type (5,6,7) was begun. The size of the radiant portion in them continually increased.

A pipe still widely used in USSR plants (Figure 30), which is a typical representative of the radiant-convection type, has been described above. In some types of pipe stills, side tubes and even floor tubes appeared, which increased the importance of radiation even more, for instance, in the pipe still depicted in Figure 32, 8, or in the Soviet pipe still shown in Figure 32, 9.

A considerable number of pipe stills have been built in our plants (the radiant-convection type with horizontal flow of combustion gases) (Figure 32, 10), which are distinguished by the long, low combustion chamber with a large number of radiant tubes (two rows on the ceiling and two rows each on the side walls), a small pyramidal convection chamber, and the movement of the gases, not over the partition wall, but a horizontal stream through a tube sheet in the partition wall.

The convection chamber was given a truncated shape because of the following considerations. The gases passing from the wall to

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the horizontal flue are cooled and their volume decreases. Consequently, their velocity also declines. In order to hold this velocity constant, and consequently also hold the coefficient of heat transfer constant, the volume of the chamber must diminish as the gases advance. This condition is fulfilled in the pipe still described.

In recent years so-called two-chamber pipe stills have appeared, i.e., pipe stills with two combustion chambers and a single convection chamber (Figure 32, 11).

In these pipe stills the radiant portion is very much expanded. For example the Soviet pipe stills are characterized by greatly developed radiant surface: from 63 to 77 percent of all heat is transferred by radiation. These pipe stills have two-row ceiling and side-wall tube banks. The efficiency of these pipe stills reaches 70 percent.

30. The Structure of Pipe Stills. Detail of the Pipe Stills

The bracing of the Pipe Still (Figure 30)

Substantial vertical steel I-columns are erected on a concrete foundation, and ceiling beams attached to them. The ceiling tubes are fastened at their ends into tube sheets called supports, which in turn are connected with the bracing of the furnace. The convection tubes are likewise fastened into tube sheets. The tubes are joined together by return bends. All the metal detail of the pipe still are then installed -- the doors, inspection ports, etc. At the conclusion of all this work the furnace work is performed: the lining of the walls, the assemblage of the

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suspension arch (Figure 34) and the roof.

The Lining of the Walls

The walls must not bear any load other than their own weight. The purpose of the lining is to prevent the pipe still from losing heat into the atmosphere. The difference between the temperatures of the inner and outer walls of the furnace reaches 1,000 degrees C, consequently, the wall must have a considerable thickness. It consists of a layer of firebrick, a layer of ordinary insulating brick, and a layer of asbestos-cement insulation. The thickness of the wall reaches 200-250 millimeters. A number of expansion joints (gaps) 35-40 millimeters wide are placed in the furnace wall with asbestos-cord packing. They allow the walls to expand on heating and contract on cooling.

Inspection ports and doors with eye holes are cut in the walls for convenient inspection of the operation of the heater. The arch of the furnace is made of shaped firebrick to protect the metal parts of the ceiling from the flames; the roof, covered with Eternit or galvanized iron, is mounted over the arch.

Tubes and Return Bends

Only seamless drawn carbon-steel tubes, or in some cases alloy steel containing chromium, molybdenum, nickel, are used in pipe stills. For pipe stills operating under high pressures the tubes are made with walls as thick as 13 millimeters.

To connect the tubes of a pipe still into a single coil,

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their ends are joined by special fittings called return bends (Figure 35). A return bend consists of a cast or forged steel detail in which two or four tubes are run. It can be taken apart for convenience of servicing. A two-tube return bend is shown in Figure 35. The ends of the tubes are run into the recess 6 and are tightly fitted into the screw-thread. The crude oil enters through one tube, makes a turn in the chamber 5, and leaves through the second tube. The ground stoppers 1 are placed in the free apertures of the return bend. The stop bolt 4 with the plate 2 is placed in the stay 3. By turning the bolt the stopper can be tightly pressed against the walls of the opening of the return bend.

There are many types of return bends. All of them must meet the following basic conditions: the method of connection must be simple, allowing easy attachment and removal during furnace repairs; their shape must be such as to create the minimum hydraulic resistance; they must be able to withstand temperatures, pressures and the corrosive action of crude oil.

The Horizontal Flue and Smokestack

For combustion gases with temperatures over 450 degrees C the horizontal flue is built of firebrick. In places where the temperature of such gases is usually lower than that, it is lined with second-grade brick. Smokestacks may be either brick or metal. In oil refineries they are usually metal. The metal pipes are reinforced by tension members to assure stability.

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IV. THE CONCEPT OF DISTILLATION AND RECTIFICATION

31. The purpose of Distillation and Rectification Processes. Methods of Distillation

Of all the processes connected with petroleum refining, the most important are those of distillation and rectification.

Distillation is the process by which crude oil may be separated into fractions: a gasoline fraction, a ligroin fraction, a kerosene fractions, etc.

The simplest method of distillation is batching, for instance, in a still. The crude is charged into the still and the prescribed amount of petroleum products distilled off. The residues are discharged and the still is again charged with crude. This operation takes considerable time and is of no advantage for the distillation process.

The method of continuous distillation is more advantageous. A certain part of the crude is continuously distilled off and is continuously replaced by the same amount of fresh crude. The gradual evaporation of the vapors from crude, for instance, in the case of batching, is called the successive-flash vaporization process for the fractions of petroleum. Another distillation method is often used in the plants: the single-flash process. This consists of heating the crude to the necessary temperature in a pipe still, then delivering it to the evaporating part of the installation where a prescribed portion is evaporated and the residue drawn off at the bottom of the evaporator.

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However, the vaporization of mixtures of volatile hydrocarbons does not complete the distillation process. The vapors must still be separated into fractions and then condensed.

The process of separating the vapors into fractions is conducted in rectification columns and is called rectification. The basic qualities of commercial petroleum products depend largely on this most essential process.

The simplest process of distillation is that of single liquids, for instance, water. When ordinary water containing various salts and sediments is distilled, pure distilled water is obtained.

A more complicated case of distillation is that of a mixture of two liquids, for instance, water and alcohol, in which pure alcohol is distilled and water is concentrated in the residue. A still more complicated case is the distillation of a mixture of many mutually soluble substances, for instance, that of petroleum or petroleum products.

32. Distillation in Vacuum and under Pressure

We have already learned that the boiling of a liquid represents a process of overcoming the external pressure by its vapor pressure of the liquid. When the external pressure is increased, the boiling point of the liquid is also increased (See Section 6), while on the other hand, when the external pressure is reduced, the boiling point of the liquid is reduced. In refinery practice, distillation is conducted either under in-

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creased pressure (as compared with atmospheric pressure) or under less than atmospheric pressure, that is, in vacuum.

One example of distillation under pressure is the stabilisation of crude oil or gasoline in stabilization plants designed to evaporate the gaseous hydrocarbons from gasoline, under pressure, and thus make it more stable.

Vacuum distillation is employed in the production of all types of lubricating oils. In this case the vacuum is necessary, since when mazut is heated to high temperatures, the heavy hydrocarbons it contains break down into smaller molecules. Distillation in vacuum allows the raw material to be heated to lower temperatures than can be done under atmospheric pressure. For instance, heptadecane, $C_{17}H_{36}$, boils at 303 degrees C under normal pressure, while when the pressure is reduced to 100 millimeters of mercury its boiling point falls to 223 degrees C and at 10 millimeters of mercury it boils at 161 degrees C (Table 7).

Table 7

Absolute pressure in mm of mercury	Boiling Point in degrees C.	
	Heptadecane	Water
760	303	100
730	--	99
100	223	51
50	201.5	38
30	187.5	29
20	177	22
10	161	11
4	--	0

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As the pressure of a vacuum approaches zero, (Table 7), it affects the boiling points of a substance in different ways. When the pressure is reduced from 760 to 730 millimeters of mercury, i.e., by 30 millimeters, the boiling point of water is reduced by only 1 degree C, from 100 to 99 degrees C. When the pressure is reduced by the same 30 millimeters, but from the residual pressure of 50 millimeters to one of 20 millimeters, the boiling point of water is reduced from 38 to 22 degrees C, i. e., by 16 degrees C, while when the pressure is further reduced by 16 millimeters (from 26 to 4 millimeters of mercury) the boiling point of water falls 22 degrees C.

The conclusion may be drawn that distillation under a residual pressure close to normal (low vacuum) gives no advantage.

To effect great reductions in the boiling point of a liquid the so-called high or deep vacuum must be employed, i.e., a pressure of 20-40 millimeters absolute of mercury.

Residual pressures ranging from 40-45 to 100-120 millimeters of mercury are ordinarily employed in refineries in the production of lubricating oils.

33. Distillation with Steam

In the distillation of petroleum, steam acts as a vacuum: by its vapor tension it reduces the total vapor tension of the mixture and thus reduces the boiling point of the higher-boiling hydrocarbons.

In the distillation of petroleum products with steam, the

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consumption of steam depends on the molecular weight of the product being distilled. Its consumption is small when distilling the low-boiling fractions, but as the molecular weight of the fraction increases, steam consumption also increases.

The steam consumption x (in percent of the distillate) is determined by the following formula:

$$x = \frac{760 - P}{P} \cdot \frac{18}{M} \cdot 100\%,$$

where P is the partial pressure of the distillate in the mixture,

M is the molecular weight of the fraction,

18 is the molecular weight of water.

Let us determine the steam consumption in the distillation of gasoline with a molecular weight of 114, at 100 degrees C. At this temperature the vapor pressure of the gasoline is 260 millimeters of mercury. Consequently,

$$x = \frac{760 - 260}{260} \cdot \frac{18}{114} \cdot 100 = 30\%.$$

At 130 degrees C the vapor pressure of the gasoline is 700 millimeters of mercury. We find the steam consumption on distillation of the gasoline at 130 degrees C:

$$x = \frac{760 - 700}{700} \cdot \frac{18}{114} \cdot 100 = 1\%.$$

Thus, by increasing the temperature of the gasoline by only 30 degrees C we reduce the steam consumption from 30 percent to 1 percent. Of course in this case it is considerably more advantageous to conduct the distillation at higher temperatures with

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a small steam consumption.

In distilling the higher-boiling fractions of petroleum, a high steam consumption is unavoidable, since otherwise the heavy hydrocarbons will be broken down at high temperatures.

Let us find the steam consumption in the distillation of motor-oil distillate with molecular weight 400, at a temperature of 300 degrees C. The partial pressure of motor-oil distillate at 300 degrees C is 40 millimeters of mercury. Consequently:

$$x = \frac{760 - 40}{40} \cdot \frac{18}{400} \cdot 100 = 81\%.$$

To use so much steam on distillation is disadvantageous, and therefore as a rule distillation of lubricating oils is conducted with steam in a vacuum. A vacuum of the order of 30-75 millimeters of mercury is ordinarily used for this purpose.

Let us compute the steam consumption for the distillation of the same motor-oil distillate, if it is conducted under a pressure of 50 millimeters of mercury at a temperature of 300 degrees C.

In this case the total pressure will be 50 millimeters, and therefore we substitute 50 millimeters for 760 millimeters in the formula:

$$x = \frac{50 - 40}{40} \cdot \frac{18}{400} \cdot 100 = 1.1\%.$$

Thus, a considerable lowering of the distillation temperature by the use of a vacuum alone would require low absolute pressures which in practice would be very difficult to attain, while the re-

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duction of distillation temperature by using steam alone would require a high steam consumption which would be very disadvantageous. The combination of moderate residual pressures (easily attainable at the plants) of the order of 40-75 millimeters of mercury with low steam consumption thus appears to be the most advantageous variant of mazut distillation.

34. The Distillation of Mixed Liquids

Each liquid forming part of a mixture tends on heating to liberate a certain amount of vapor, with the lower-boiling liquids liberating more vapor than the higher-boiling liquids.

When several liquids are mixed (for instance, hydrocarbons), each of them keeps its own property of emitting vapor at a given temperature. If a mixture contains equal quantities of two hydrocarbons, one of which is low-boiling and the other high-boiling, the low-boiling hydrocarbon will liberate more vapor. However, if a mixture contains two liquids boiling at the same temperature, the liquid contained in larger quantity will evaporate a larger amount of vapor.

Consequently, the evaporation of any liquid depends, in the first place, on its vapor tension (which in turn depends on its boiling point), and, in the second place, on the proportion of the mixture that it constitutes.

35. The Concept of Rectification

Petroleum cannot be separated into fractions by simple distillation. Assume that we are distilling a mixture of benzol and toluol

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in an Engler still. In spite of the fact that the boiling point of benzol is 80.4 degrees C and that of toluol is 110.6 degrees C, toluol passes over with the very first drop, and the very last drop of the distillate will still contain benzol mixed with toluol. Only at the low initial temperatures will benzol be the chief component of the distilled mixture, while toluol is evaporated only in small quantities. As the temperature rises, the percentage of benzol falls and that of toluol increases. Towards the end of the distillation the main component becomes toluol, while benzol will be contained in the mixture in small quantities.

Let us assume that we are distilling a mixture of 50 percent benzol and 50 percent toluol, and take six fractions, with toluol and benzol distributed in these fractions in the following proportions:

I	fraction contains	94%	benzol	and	6%	toluol
II	" " "	80%	"	"	20%	" "
III	" " "	60%	"		40%	" "
IV	" " "	40%	"	"	60%	" "
V	" " "	20%	"	"	80%	" "
VI	" " "	6%	"	"	94%	" "

On mixing the first three fractions we obtain a mixture containing 78 percent of benzol and 22 percent of toluol, since $\frac{94 + 80 + 60}{3} = 78$, while $\frac{6 + 20 + 40}{3} = 22$. We take a number of fractions from this mixture again on the succeeding distillation. Then we again mix the fractions richest in benzol and finally obtain

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a mixture consisting, say, of 90 percent benzol and 10 percent toluol. On repeating this operation a number of times, we shall ultimately obtain almost pure benzol in the distillate. We do the same with the fractions richest in toluol, and then obtain pure toluol.

Thus, we successively and repeatedly perform the processes of evaporation and condensation, and with each new process of evaporation we obtain a purer and purer substance in the vapor phase. This prolonged process may be conducted by a single method in an apparatus known as a rectification column.

Let us consider the operation of a Gadaskin laboratory rectification column (Figure 36).

The distillation is performed from the metal still 1, which is provided with the metal Gadaskin column 2. The diameter this column is 5 centimeter and its height is 50 centimeters. At the upper constricted part of the column the so-called dephlegmator 3 is attached. The rest of the lay-out is analogous to that of the Engler still (thermometer 4, cooler 5 and receiver 6).

The Gadaskin column is filled with aluminum rings or glass balls. The column is therefore termed a packed dolumn. It is insulated with asbestos to avoid sudden chilling. The still is charged with liquid (for instance with gasoline) and heated by a burner. The gasoline vapor, on meeting the packing in the column, is divided into numerous fine jets. This cools them off slightly, and the heavier fractions condense and flow back into the still.

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This condensate is termed phlegma. The remainder of the vapor is enriched in the lighter hydrocarbons. The higher the vapor rises in the column, the more of the high-boiling hydrocarbons are condensed, and the richer the vapors become in the lighter components. Finally, the vapors leave the column and enter the dephlegmator. Here still another portion of the high-boiling hydrocarbons is condensed, while the vapors leaving the dephlegmator are routed to the cooler, and then, on being cooled, pass to the received in the form of condensate. As we see, repeated condensation of the heavy fractions takes place along the entire length of the column, resulting in enrichment of the vapors in the lighter fractions as we approach the top of the column.

V. THE LAY-OUT AND OPERATION OF RECTIFICATION COLUMNS

36. Bubble-Cap Rectification Columns.

Two types of columns are used in refineries: the packed and the bubble-cap type.

Packed columns (see Figure 40, a) like the laboratory columns, are provided with packing consisting of clay rings ranging in size from 6 x 6 to 75 x 75 millimeters (Figure 37). Packed columns are used today only in the oldest units and are no longer constructed in new units.

Bubble-cap columns have transverse baffles termed plates. The plates have apertures with the riser 1 welded in. Each riser has a special cap 2 with slots on the bottom. There are a few dozen, sometimes even hundreds, of such caps on each plate (only

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one cap is shown on Figure 38, for the sake of simplicity). The plate is provided with the downcomer 3, through which the liquid flows from the upper plate to the lower. The top of the downcomer projects a few centimeters above the plate, and therefore a layer of liquid of constant depth is formed on the plate. The lower ends of the downcomers are immersed in the layer of liquid on the next lower plate, so that the vapors do not enter them. The plates operate as follows. The caps are solid and have slots which are covered with liquid, on their bottoms. The vapors therefore traverse this liquid, or, as they say, bubble through it, before leaving the plate.

What happens to the vapors and liquid during their contact on the plate? The temperature of the liquid on the upper plate is lower than that of the vapors rising from the lower plate. Therefore the lighter fractions contained in the liquid are evaporated by the heat of the incoming vapors, while the high-boiling fractions contained in the vapors are condensed by contact with the colder liquid and remain in it. Thus, the processes of evaporation of the low-boiling fractions and condensation of the high-boiling fractions take place simultaneously on each plate. The vapors that pass to the next plate are lighter than when they arrived at the preceding plate. As they ascend from plate to plate, the vapors become more and more enriched in the low-boiling component, and, given a sufficient number of plates, vapor of the required composition may be obtained at the top of the column.

Rectification thus consists of a process of repeated evaporation and condensation.

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The rectification of petroleum is a very complex process. We shall therefore first consider two of the simplest cases of distillation: that of a two-component mixture of benzol and toluol, and that of a triple mixture of benzol, toluol and xylol (Figure 39).

Assume that we must separate a mixture of benzol, B, and toluol, T. This mixture, heated to the necessary temperature, is supplied to the middle part of the column 1 (see Figure 39). The vapor ascends in the column, and practically pure benzol emerges at the top. The benzol vapor is condensed in the condenser 2 and cooled in the cooler 3. Part of the benzol enters its container, while part of it is returned to the column in the form of a reflux, which is necessary to remove the excess heat from the vapors.

The liquid flows to the bottom of the column, which is similarly provided with plates. On these plates rectification occurs, but here not of vapors, as on the top of the column, but of liquid, to free it from benzol. As the liquid flows downward, its benzol content becomes less and less and practically pure toluol emerges at the bottom of the column.

Additional heat must be brought in to distill off the benzol. For this purpose the lower part of the column is provided with the steam coil 4. Thus, the upper part of the column is designed to rectify vapor and is termed the rectifying section while the lower part is designed to rectify liquid and is termed the stripping section.

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It is entirely natural that the temperature of the liquid in the stripping section of the column should be close to the boiling point of toluol, 110 degrees C, and that on the top it should approach the boiling point of benzol, 80 degrees C.

Rectification of a triple mixture is conducted in two columns and proceeds in the following manner (see Figure 39). In the first column 5 (from the bottom) the xylol is drawn off, while at the top a mixture of toluol and benzol emerges. This mixture is then introduced into the second column, where the process proceeds in entire analogy to the above-described one.

Three columns would be required to rectify four components. Thus, the number of columns required to rectify n components is n - 1. In practice this number of columns is not constructed. Ordinarily a single column is constructed, which represents, as it were, several superimposed columns.

Figure 40, b represents a rectifying cap column consisting of four sections. The crude enters the middle part 1 of the first and lowest section, where the gasoline, ligroin kerosene and solar oil are distilled off, and mazut 2 issues from the bottom of the column. The mixture of vapors of petroleum products passes through the constricted part 3 of the column to reach the II section, where the solar oil is separated and flows out through the bottom of section I, while the mixture of gasoline, ligroin and kerosene is routed to section III. Here kerosene, 5, is taken off in the form of phlegma, while the mixture of gasoline and ligroin vapors enters the last section IV of the column, where the

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ligroin is separated and emerges from the column in the form of the phlegma 6, and the gasoline passes through the top of the column first into the condenser then into the cooler and finally into its receiver. Part of the gasoline is returned to the column at 7 for refluxing.

The role of refluxing. Refluxing is one of the most important factors in regulating the operation of a column. It is done from the top of the column and meets the oncoming vapor moving countercurrent. By taking up some of the heat of that vapor, it facilitates the condensation of the higher-boiling components, i.e., it plays the same role here as the plates do. Ordinarily, refluxing takes up a very large amount of heat from the vapors (up to some millions of large calories per hour) and thus replaces a considerable number of plates.

Refluxing covers the entire height of the column (in the rectifying section), although it is only introduced at one or two points. For instance, in the column described, gasoline is injected at point 7 to reflux the ligroin section. This reflux causes the condensation of the ligroin and some of the gasoline as well. The mixture of gasoline and ligroin flowing off through pipe 8, serves as the reflux for the kerosene section III. The mixture of gasoline, ligroin and kerosene, flowing off through pipe 9, serves as the reflux for section II, and finally the mixture of all four components serves as the reflux for Section I. In all cases the lower-boiling component appears in the role of the reflux.

There are three methods of refluxing (Figure 41):

(a) cold, or sharp,

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(b) hot, or dull,

(c) circulating.

Cold refluxing consists in pumping a sufficient quantity of the cooled distillate withdrawn from the top of the column back as a spray into the top of the column. On contact of the cold spray with the vapor of the petroleum product, the following processes take place: condensation of the high-boiling components of the vapor and heating and evaporation of the spray on account of the heat of condensation. The vapor from the spray together with the vapor of the volatile component pass into the condenser. The subsequent path of the vapors is described above. The high-boiling component, separated on the condensation of the vapors is returned to the first plate (the first from the top). The excess liquid formed on the first plate flows off to the second plate and serves as reflux for the vapors ascending from that second plate, the excess of liquid formed on the second plate flows off to the third plate, and so on.

In this manner the introduction of the reflux at the top of the column produces a reflux throughout the entire column.

Hot (or dull) refluxing is effected as follows. A partial condenser in the form of a pipe coil is installed on top of the column and water or raw material is pumped through it. In the latter case, part of the heat of condensation of the vapors is usefully employed to preheat the raw material. The vapors of the high-boiling hydrocarbons are condensed in the partial condenser and flow off in the form of hot liquid onto the upper

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plate, producing a reflux for the lower plates in the same way as with cold spraying. It is clear that in this case more liquid is needed than with cold refluxing.

A drawback of the hot reflux method is the complexity of the condenser design and its liability to corrosion, as a result of which leaks may occur and the distillates may be spoiled by the entrance of raw material into the column.

The circulating reflux method consists in the withdrawal of liquid from a number of plates and in pumping it back, through heat exchangers, onto the upper plates. This is the most advantageous method of refluxing, since it allows utilization of a considerable amount of heat for preheating raw material in the heat exchangers.

The stakhanovites of the refineries have reduced the fuel consumption 15-25 percent on changing over the columns to the circulating reflux system. At the same time they increased the productivity of the units since by preheating the raw material in this way they freed the capacity of a pipe still which could then be used to preheat an additional quantity of raw material.

The amount may range from twice to five times the amount of the distillate obtained.

Superheated steam is fed into the stripping section of the column to vaporize the volatile components still remaining in the mazut.

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37. The Efficiency of Separation of the Distillates and the Completeness of Their Withdrawal

The efficiency of rectification is determined by the content in a given distillate of other fractions boiling below or above the boiling point of the fraction in question. For instance, in rectifying a triple mixture of benzol, toluol and xylol, the efficiency of rectification of toluol will be determined by the absence of benzol and xylol in it.

The efficiency of rectification depends on the number of plates in the column and on the amount of the reflux. The more plates there are in the column, and the more refluxing is done in it, the more efficiently will it operate.

The efficiency of rectification of light petroleum products is determined, by general agreement, by Engler distillation.

Figure 42 gives two cases of separation of ligroin from gasoline. The initial boiling point of gasoline is designated by t_I and its end point by t_E; the initial boiling point of ligroin is designated by t'_I and its end point by t'_E. In the first case (I) the initial boiling point of ligroin t'_I is higher than the end point of gasoline t_E by the value $t'_I - t_E$. In the second case (II) the initial boiling point of ligroin t'_I is lower than the end point of gasoline t_E by the value $t_E - t'_I$. There takes place, as they say an overlapping of the fractions. This column operates poorly and inefficiently.

The completeness of recovery of the fractions is closely

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connected with the sharpness of rectification. If a column is not operating sharply, the fractions will overlap and part of the gasoline will remain in the ligroin, part of the ligroin in the kerosene, and part of the kerosene in the mazut.

Table 8 shows recovery of a gasoline-ligroin fraction of 23.5 percent (including 14.5 percent of gasoline in Unit II up to 215 degrees C, and of 21.5 percent in Unit I up to 220 degrees C. Consequently, the column of Unit II is operating more sharply than that of Unit I.

Table 8

	Unit I	Unit II
Light gasoline	to 175 degrees, 14.5%	to 157 degrees, 14.5%
Ligroin	to 220 degrees, 7.0%	to 215 degrees, 9%
Kerosene	to 325 degrees, 23%	to 315 degrees, 19.5%

To improve the sharpness of rectification of distillates, auxiliary stripping columns are used. These are columns with a small number of plates, to which the distillates are routed from the main column for supplementary stripping of the volatile components, for which superheated steam is fed into the stripping columns.

The number of plates in the various types of columns and stripping columns varies within a wide range (Table 9).

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Table 9

	Number of plates	
	Rectifying section	Stripping columns
For separating gasoline from ligroin	5-12	3-7
For separating ligroin from kerosene	4-8	2-6
For separating kerosene from solar oil	4-9	2-6
For separating the lubricating oil distillates	2-6	2-4

Columns with a small number of plates are used for rectification of lubricating oil distillates, since there is no need for particular sharpness of rectification.

38. The Lay-Out and Equipment of Columns

A column consists of a cylinder of which height and diameter depend on the type of column and the productivity of the unit. Thus, an atmospheric column in one of the installations is 36.5 meters high and 2.5 meters in diameter, while a vacuum column of another installation is 8.7 meters high and 6.4 meters in diameter. Plates are arranged inside the columns and consist of transverse baffles with openings provided with caps.

Columns of different types differ mainly in the design of the plates and caps. A considerable number of cap types are known, differing in the design and method of attachment of the caps themselves, in their size, shape and distribution, in the material of which they are made and in the shape and location of the downcomers.

The caps may be rectangular, round or hexagonal. The number

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of caps on a plate varies from a few to a few hundred, depending on the type of column and size of the caps.

Let us consider a few types of plates and caps.

Figure 43 shows a cast-iron plate consisting of four segments. The caps are of steel, rectangular in shape, with serrated edges. They are attached to the plate by bolts. A shortcoming of caps of this type is that the vapor bubbling through the liquid is unable to divide into small jets.

The next type of plate is steel, with steel or cast-iron caps. The cap is 107 millimeters high and 103 millimeters in diameter (Figure 44). It has narrow slots at the bottom through which the vapor passes. They are set up over the entire plate area.

Figure 45 shows a steel plate with grooved caps. In the middle quarter of the plate the grooves are fastened at some distance from each other (so as to form a narrow passage). On each two channels another similar channel with teeth is fastened.

This plate operates well and produces a vigorous atomization of the vapors into jets. (Figure 45,c).

The plate used in the column of the Winkler-Koch cracking plant, is of steel, with hexagonal cast-iron caps (Figure 46). The caps have slots on the bottom. Halfway to the top of each there are small apertures with slots to provide supplementary surface for building.

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The Winkler-Koch column is mainly used in cracking plants. Its operation cannot be considered satisfactory.

Layout and distribution of the caps must be such as to divide the vapors passing through the slots into the largest possible number of jets. In that case the vapors will have a large surface of contact with the liquid. Small caps with a large number of slots (Figure 44) satisfy this requirement.

The efficiency of a plate is equal to the ratio of the theoretically computed number of plates to their actual number used in practice. If the efficiency of a plate is 0.5, it follows that in practice two plates do the work that theoretically one would do. Plate efficiency ranges in practice from 0.2 to 0.7 (for different types of columns).

Head Tube

The column is provided with a wide curved head tube of diameter up to 500 millimeters, according to the capacity of the installation, to conduct the vapors into the condenser. If a still greater cross section is required, two head tubes are used.

Manholes

To provide access to the interior of the column for inspection and repair there are manholes so arranged that each plate can be reached. To prevent loss of the product through the manholes during operations, a special packing that withstands high temperatures is installed between the cover and the manhole. The cover is attached to the manhole by steel bolts.

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Equipment of Vacuum Columns

A vacuum reaching 30-75 millimeters absolute pressure is maintained in vacuum columns.

The vacuum is created by two methods: (1) by the condensation of steam and of part of the hydrocarbons leaving the top of the column and (2) by pumping out the uncondensable gases.

The condensation of vapor is effected in what are called barometric condensers (Figure 47) which operate in the following way. The vapor 1, on meeting the water 2 in the condenser, is condensed. A vacuum is formed by the sharp reduction in the volume of the vapor. The uncondensable gases and air are pumped out by a steam-jet ejector (Figure 48) or a vacuum pump.

The vacuum ejector works on the following scheme. Steam enters the nozzle 1 through the admission duct 1. There it meets the vapor 3 coming from the barometric condenser and draws them at high speed through the constricted part 5, where the gases are compressed; the more strongly they are compressed, the higher the degree of rarefaction attained in the column.

Assume that a sevenfold compression is effected in the ejector. What will then be the rarefaction in the column?

The gases are ejected into the atmosphere, which has a pressure of 760 millimeters of mercury. An absolute pressure of 760 : 7 = 109 millimeters of mercury may be created in the ejector.

By the installation of a second such ejector the absolute pressure in the column may be reduced to 100 : 7 = 15 millimeters

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of mercury, since the second ejector ejects vapor into the first one.

In practice, two-stage ejectors are installed in the plants and vacuum pumps are also used to create a vacuum there.

The Insulation of the Columns

To avoid heat losses the columns are insulated with a layer of insulating material up to 100 millimeters in thickness.

A well-insulated column loses no more than 2 - 3 percent of the heat.

Determination of the Number of Plates Used In Practice

The theoretical number of plates is determined by calculation. On selecting the type of column, the number of plates required in practice is determined. Assume that we have chosen a type of column and that the number of plates has been theoretically fixed at 10. The efficiency of the plates of this column is 0.5. The number of plates required in practice will then be 10 : 0.5 = 20.

The distance between the plates, depending on the velocity of the vapors and the designs of the plates, ranges from 250 to 600 millimeters. The vapors in their travel may entrain droplets of liquid. If the distance between the plates is small, droplets of liquid will spout from the lower plates to the upper ones, considerably impairing the operation of rectification. The higher the velocity of the vapors and the less the distance between the plates, the greater the spouting will be.

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For example, in an atmospheric column with a distance of 0.45 meters between the plates, an increase in vapor velocity from 0.6 to 1.7 meters per second will increase spouting to 21 percent.

Velocity of vapors	% Spouting
0.6 meter/second	traces
1.2 meter/second	7
1.7 meter/second	21

Consequently, there is a limiting vapor velocity for each column, which cannot be exceeded without impairing the sharpness of separation.

For atmospheric columns the vapor velocity is taken as 0.7 to 0.9 meter/second, and for vacuum columns up to 1.2 meter/second.

In fixing the height of the column the number of plates and the distance between them is taken into consideration. Assume that there are 30 plates in a column, with a distance of 0.4 meter between them. The lower and upper parts of the column (without the plates) are taken as equal to 1.2 meters.

The height of the column $H = 30 \times 0.4 - 1.2 \times 2 = 14.4$ meters.

The diameter of the column depends on the quantity of vapor passing through it and on its velocity, and ranges from 3 to 4 meters for atmospheric columns and from 6 to 7 meters for vacuum columns.

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VI. CONTROL INSTRUMENTS

Oil refineries are provided with various control instruments for measuring temperatures, pressures, levels, quantities or volumes of liquids and vapors, and so on. Besides these instruments, oil refineries also use recording instruments, since it is very important to know how the prescribed operating conditions being followed at each moment of operation.

To create a smooth and constant regime for the installations, automatic instruments are employed to regulate temperatures, pressures and levels at the most vital points of the equipment.

For convenience of control and surveillance over the operation of the equipment, all the principal control and metering instruments are installed in a single place, on the control panel in the operating room. The instruments are connected with the equipment by tubes and electric wires. Figure 49 shows the control panel of one of the modern oil-refining installations.

39. The Measurement of Temperatures

The simplest instrument for measuring temperature is the thermometer. However, the thermometer is only suitable for measuring temperatures no higher than 360 degrees C and then only directly at the equipment.

The measurement of higher temperatures is effected by the aid of a pyrometer, a device consisting of two parts, a thermocouple and a galvanometer. The pyrometer utilizes the following phenomenon, well known in physics: when the junction 3 of two

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different metals is heated, what is termed thermoelectricity is produced, and may be measured if the cold ends 2 of the wires are connected to the galvanometer 4. For each definite temperature to which the given thermocouple is heated, the galvanometer needle gives an entirely definite deflection, according to which the scale of the galvanometer is calibrated with temperature readings. The thermocouples are placed in those points of the equipment where the temperature must be measured, while the galvanometer (one galvanometer can serve a number of thermocouples) is installed on the control panel in the operating room; the cold ends of the thermocouple are connected to the galvanometer by the wires 1. Pyrometers may be indicating (as we have just described in detail), recording, and regulating (automatic thermostats).

By connecting the needle of a galvanometer with a pen and placing paper under it, which is moved by a clock mechanism, we obtain a recording pyrometer (Figure 51). The temperature of an apparatus at any moment during operation of an installation may be determined from the record made by the instrument.

The most important points are automatically regulated at installations (temperature at the top of the column, temperature of the combustion gases and of the partition wall, temperature of feed at pipe still outlet. For instance, by the aid of a special arrangement a pyrometer is connected with the reflux pump and automatically increases the amount of the reflux if the temperature rises at the top of the column. This device operates as follows. In consequence of the increase of the reflux delivered to the column, the temperature at the top of the column falls, and,

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when the galvanometer needle reaches a predetermined lower limit, the reflux is automatically reduced (since the number of strokes of the spray pump is automatically reduced), and the temperature of the top of the column will likewise increase up to a predetermined upper limit. In this way the temperature will always fluctuate between two assigned limits, an upper and a lower. The difference between these two temperatures is usually 5 to 10 degrees C.

40. The Measurement of Pressure

Pressure is measured by manometers. The pressure readings for equipment are one of the basic indices, since each apparatus is intended to operate at a definite pressure, which it is forbidden to exceed. Therefore the manometers are checked by a special organization. It is not allowed to work with manometers that are not sealed by this organization.

Tube manometers (Figure 52) are the type used most in the petroleum industry. The main part of a manometer of this type consists of a bronze or steel tube with an oval cross section. The free end of the tube is sealed and connected with a mechanism and pointer; the other end is connected to a connecting pipe with a screw thread. When the manometer is connected to an apparatus under pressure, the tube tends to bulge outward. Thereupon the pointer connected to the free end of the tube deviates from its original position. The higher the pressure in the apparatus, the greater the deviation of the pointer. The face of the manometer is calibrated in accordance with the various pressures. Depending on their purpose, manometers are manufactured and calibrated for various pressures, ranging from 0.5 atmosphere to tens

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and even hundreds of atmospheres.

Manometers are installed at places accessible to observation or on the control panel in the operating room. The proper manometer is selected for each apparatus. For instance, a manometer with a maximum pressure of 12 atmospheres is installed on an apparatus working at a pressure of 8 atmospheres. Oil refineries are also provided with recording manometers.

41. Level Indicators. Level Regulators. Meters

The simplest instrument for measuring the level of a liquid is the water gauge. The water gauge is installed so that its lower part is in contact with the liquid and its upper part is in contact with the vapor space of the apparatus. It is used mainly on boiler installations.

Float indicators are used to measure the level of liquid in reservoirs (Figure 53). A float, floating on the surface of the liquid, is connected by a band passed over the top of the reservoir with a counterweight which moves along a graduated rod from which the level is read off.

In apparatus operating under pressure and at high temperatures (in evaporators or columns) the level of the liquid is checked by the use of test-cocks located at various heights.

The purpose of level regulators is to regulate the level of the liquid in columns, water separators, etc. A liquid-level regulator is shown in Figure 54. The float chamber 1 is connected with the apparatus by means of connecting pipes. A ball float,

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rising and falling with the liquid, is placed in the chamber. The motion of the float is transmitted by the rod, the system of levers and the link 2 to the valve 7, which regulates the quantity of liquid flowing through the pipe A.

At the present time level regulators with pneumatic control are in widespread use.

Special meters are used to gauge the quantity of liquid, vapor or gas flowing through a piep line. Dry and wet gas meters are used to determine the volume of gas. Dry meters consist of a bellows of determinate volume, which is alternately filled with gas and deflated. Wet meters consist of a metal barrel half filled with water. Four baffles, dividing the barrel into four compartments, rotate within it on a spindle. The number of rotations of the barrel is registered by a meter.

Meters for measuring liquid are similar to the wet meters for gas.

Consumption meters, which are widely used in oil refineries, determine the amount of liquid passing on the basis of the fall (or differential) in pressure. An orifice plate with an aperture less than the diameter of the piping is installed between flanges of a coupling. As a result of the reduction in the diameter of the line at the place the orifice plate is installed, the velocity of passage of the substance is increased and the pressure rises. A special device termed a differential manometer measures the difference of pressure (pressure drop) before and after the diaphragm. Knowing the pressure drop, the quantity of substance passing through the piping in unit time may be computed.

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CHAPTER SIX

THE PREPARATION OF CRUDE OIL FOR REFINING

The crude oil should arrive at the refinery only after careful examination and preparation, consisting of grading, dehydration and demulsification.

The crude as it leaves the well contains water and sediment, and sometimes it is in the form of an emulsion. On the way to the plant, and when stored under unsatisfactory conditions (for instance, in open earthen sumps) crude may be mixed with still more water and sediment.

For this reason measures must be taken to make it possible to deliver crude from the production lot to the refinery without admixture of water or sediment. The mixture of different crudes is likewise inadmissible, for it may adversely affect the quality of the distillates. For instance a crude containing a fraction of high-grade transformer oil becomes unsuitable for producing that oil if mixed with a small amount of paraffin crude, since this would increase the freezing point of the distillate. Careful surveillance over the quality of the crude is therefore necessary during the entire trip from the oilfield to the refinery.

42. The Grading of Crudes

In one and the same region where crudes are produced from different horizons they not infrequently differ sharply in their qualities. Their mixing during production or transportation to the refinery lowers the quality of the distillates.

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We have just given an example of the spoilage of crude containing transformer oil. Let us take a second example. Heavy Malgobek crude contains 1 percent high-octane gasoline, while light Malgobek crude contains 20 percent low-octane gasoline. If these crudes are mixed, the high-octane gasoline of the heavy crude is irretrievably lost. Therefore the high-quality crudes, or, as they are called, the graded crudes, must be protected from mixing with low grade crudes, and crude must be kept track of from the well where it is produced until it reaches the distilling plant.

Graded crudes must be kept in separate reservoirs at the producing lot.

When crude is being transported to the plant, especially over long distances, care must be taken to avoid mixture of graded crude with ungraded.

43. The Effect of Contamination of Crude on its Refining

As indicated in the last Section, the crude as produced from the well is ordinarily contaminated by salts, mechanical admixtures and water, and is sometimes in the form of an emulsion, i.e., so stable a mixture of oil and water that it will not separate into layers on mere standing.

In some crudes the water content may run as high as 25-35 percent and even up to 60 percent, being particularly high in winter. The sediment in crude reaches 0.2 - 0.3 percent, while in salts it runs up to a few thousand milligrams per liter.

Water, sediment and salts exert a harmful influence both on the process of distillation and on the equipment used for it.

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The Influence of water

A high water content in crude causes the following undesirable phenomena:

the productivity of the installation is reduced, since the volume of a given quantity of steam is 15 times that of the corresponding amount of petroleum vapors;

the fuel consumption is increased, since it is unproductively expended on evaporating water;

the water consumption for condensing and cooling the vapors is increased, and at the same time the condensation of gasoline vapors is impaired, because the surface of the condensers and coolers is not figured for cooling the excess amount of steam;

rectification is impaired, since as a result of the increase in the total quantity of vapor in the column (owing to the water) the velocity of the vapors is increased, which interferes with the operating conditions of the column;

the pressure in the equipment is increased several times, which may lead to explosions in the apparatus.

The Influence of Sediment

Clay, sand and suspended salts lead to: wear on the equipment by the mechanical grinding of the sediment; fouling of the equipment, especially in the tubes of the heat exchangers and pipe still heaters; impairment of the heat transfer and increasing the fuel consumption; premature shutdown of the unit owing to fouling or burning of the tubes.

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The Influence of Salts

The harmful influence of salts is expressed in corrosion of the equipment. Hydrochloric acid is formed from the chlorides contained in crude, and vigorously attacks metals, especially at the moment of condensation. For instance, on refining Ishimbay crude at one plant, the condensers and coolers were corroded and went out of commission after a few dozen hours of operation. The upper parts of the cribble towers were also badly damaged by corrosion. After 15-20 days of operation the pipe-still tubes proved to be corroded to a depth of 1 millimeter. Besides producing corrosion, the harmful influence of salts is also manifested in fouling the equipment.

Consequently crude should arrive at the refinery after preliminary settling and removal of water, sediment and salts.

44. Oil Emulsions and Methods of Breaking Them

An emulsion is a mixture of two mutually insoluble liquids in which one liquid is distributed through the other in the form of minute droplets (in a suspended state).

In external appearance emulsions are opaque, uniform liquids from white to dark color. The viscosity of an emulsion increases with its water content. It may be creamy consistency or even be paste-like.

Milk may be cited as an example of an emulsion known to everyone: minute particles of organic (fatty) substances are distributed through water.

Crude-oil emulsions may be of two kinds: (1) oil in water, with minute oil particles distributed through water; (2) water in oil,

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with minute water particles distributed through oil.

An emulsion is stable when each particle of one liquid contained (in finely divided state) in the other is covered by a film of a substance which is called an emulsifier. In the absence of such a substance the particles of the liquid will coalesce and the emulsion will separate into two layers, oil and water respectively.

The sodium and calcium salts of the naphthenic acids, asphaltic and resinous substances, etc, may act as emulsifiers.

There are two types of emulsifiers. The first type is readily soluble in water and very slightly soluble in petroleum. Such emulsifiers are termed hydrophile, i.e., loving an aqueous medium (sodium salts of naphthenic acids and sulfonic acids). The second type of emulsifiers, on the contrary, is readily soluble in oil and poorly soluble in water. Such emulsifiers are termed hydrophobe, i.e., not loving water (calcium salts of naphthenic acids, resinous and asphaltic substances, etc). If the water particles are distributed in water, the emulsion is called hydrophile (Figure 55).

Hydrophobe emulsions can be dissolved in crude oil or petroleum products, since oil is the external phase; hydrophile emulsions can be dissolved in water, since water is its external phase.

To determine the type of an emulsion, a small amount of it is dissolved in water and in gasoline. If it dissolves in gasoline, it is hydrophobe; if it dissolves in water, it is hydrophile. Most petroleum emulsions are hydrophobe.

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Methods of Breaking Emulsions (Demulsification)

To break an emulsion the protective film that surrounds the finely divided particles of liquid must be destroyed. This may be effected by a number of methods: (a) heating and standing, (b) use of demulsifiers, (c) electrical methods.

Dehydration and Demulsification of Crudes by Heating and Standing

When an emulsion is heated the film of emulsifier expands and bursts, and the particles of liquid coalesce. The water settles to the bottom, the oil rises to the top. To break an emulsion it is sometimes enough to heat it to 50-60 degrees Centigrade, but in some cases emulsions are not broken even on heating to 100-120 degrees Centigrade.

The settling and preheating of watered or emulsified crudes may be performed in metal settling tanks. The crude in such a tank is heated by steam coils for 24 hours and longer, but heating to over 50-60 degrees Centigrade is not recommended, since loss of the light fractions may result.

A widely used method of dehydrating crude is by the use of horizontal cylindrical apparatus forming a part of the distilling installation and termed water and sediment separators (Figure 56). The crude is withdrawn from the reservoir 1 and is pumped through the heat exchanger 3, in which it is heated to 100-170 degrees Centigrade and then enters the water and sediment separator 4. Water is continuously drawn off from the bottom 7 of this separator, while the oil separated by standing emerges from the top. By this method

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of dehydration the settling time is shortened to 1-2 hours and the losses of the light fractions is greatly reduced.

This method is very convenient for dehydrating unstable petroleum emulsions. Other methods are employed, however, for dehydrating and breaking stable emulsions.

Breaking Emulsions and Desalting Crudes By Using Special Substances.

A number of substances have an action directly opposed to that of emulsifiers. Let us assume that a mixture of oil and water contains a hydrophile emulsifier, the sodium salt of naphthenic acids, which tends to form an emulsion of oil in water. We add to the mixture the necessary quantity of a hydrophobe emulsifier having the opposite action, for example, resinous substances or the calcium salts of naphthenic acids, which tend to form an emulsion of water in oil. As a result of the contrary actions of these substances, no emulsion at all is formed, since the second emulsifier is a demulsifier with respect to the first.

The principal demulsifiers used to break petroleum emulsions are: (1) neutralized black contact (NBK), (2) various salts of naphthenic acids (for instance, aluminum naphthenate, (3) organic acids, (4) sulfonated oil (SUO).

Installations Employing NBK

When fuming sulfuric acid acts on kerosene or gas oil, sulfonic acids -- the commercial contact of Petrov -- are formed. On treatment of this product with alkali, neutralized black contact, containing at least 10 percent sulfonic acids and up to 90 percent water, is produced.

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The NBK demulsifying plant at one of the producing lots operates as follows. From the reservoir 2 (Figure 57) the emulsion is pumped into the pipe still 4. After its delivery from the supply pipe to the pipe still, 0.5 to 0.6 percent of NBK is added to the pipe under atmospheric pressure. The mixture is heated to 60-70 degrees Centigrade, and then routed to the settling tank 5. As a result of the operation of the demulsifying plant the water content is reduced from 65 percent to 2 percent. Treatment of Ishimbay crude reduces its water content from 3-3.5 percent and its salt content from 4000-4500 milligram liter to 500-700 milligram liter. Although this is a great reduction of salt, it is still not enough for normal operation of a refinery, for the salt content of crude must not exceed 75-100 milligram liter.

Aluminum naphthenate in the amount of 0.15 - 0.20 percent for 24 hours at 20 degrees Centigrade easily breaks an oil emulsion and is used in cases where NBK is ineffective.

Sulfonated oil, SUO, is very effective: 0.20 to 0.03 percent of this reagent suffices to break an oil emulsion completely. However it is very expensive and also requires the use of vegetable oil for its manufacture.

Breaking Emulsions and Desalting Crudes By Electrical Methods

In recent years electrical methods of demulsification have gained ground. They consist in the passage of a high-tension current through the emulsion in a special apparatus.

The installation pictured on Figure 58 is the most widely employed of this type. In a mixer the crude is mixed with wash water (5-50 percent), heated in the heat exchanger 3 to 60-90 degrees

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Centigrade, and then routed to the electric dehydrator 1.

In the electric dehydrator (figure 59) the emulsifier film is broken down under the action of electrical charges and the water droplets settle and are drawn off. The final layer of crude at the top is routed to the settling tank 2.

In these installations 95 percent of the water and 95 percent of the salt is removed from the crude.

Experimental installations of the TsIATIM and of Slonim have been installed in USSR plants, and have given very good results in operation. Thus, 90-97 percent desalting is attained in the Slonim installation.

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CHAPTER SEVEN

THE DISTILLATION OF CRUDE OIL AND MAZUT

Of the cycle of manifold processes performed in oil refineries, the first and most fundamental is the process of distillation of petroleum. The quality of the commercial products placed on the market depends to a considerable extent on the sharpness of separation with which the distilling installations operate.

Regardless of its type, every oil-distillation unit is designed to effectuate the following processes:

(1) pumping the raw material into the unit,

(2) heating the raw material to the necessary temperature,

(3) evaporating and fractionating the petroleum into distillates,

(4) condensing and cooling the distillates.

At the very beginning of oil refining, petroleum was distilled only in shell-still units (Figure 60). The heating equipment for these installations consisted of retorts, and the fractionating equipment of simple dephlegmators. The modern process of distilling petroleum is effected in pipe-still units. Here the petroleum is heated in pipe stills and fractionated in great rectification columns.

According to the form of pressure employed, distilling plants are divided into atmospheric and vacuum. In atmospheric plants crude oil is distilled and light petroleum products are obtained -- gasoline, ligroin, kerosene, Diesel fuel, and mazut in the residue. In vacuum plants mazut is distilled and lubricating oil distillates obtained, with mazut pitch in the residue.

Let us consider the following distilling plants:

(1) atmospheric and vacuum shell-still units;

(2) atmospheric pipe stills

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(3) vacuum pipe stills.

I. ATMOSPHERIC AND VACUUM SHELL-STILL PLANTS

45. The Batch Petroleum Distillation Plant

The simplest plant for distilling crude oil is the batch still plant (Figure 61). It consists of the preheater 1, the still 2, the dephlegmator 3, the condenser 4, the cooler 5, the scrubber for recovering gasoline from the gases 6, and the cut house 7.

The principal part of the installation is the shell still (Figure 62). This is a horizontal boiler of iron plates 10-12 millimeters thick riveted together. The still is 6-10 meters long and 2-3.5 meters in diameter. To increase the heating surface, one or two fire tubes are installed inside the still; inside the fire tubes there are burners and a combustion process takes place. The still is lined with a brick lining so that its inside lateral surfaces are bathed by the combustion gases traveling along the following route: the gases travel along flue I to the end of the still, then divide into two streams and are returned along the passage II, bathing the inside lateral surfaces of the still. When they reach the front edge of the still, the gases then proceed along passage III and bathe the lower inside surface of the still; when they reach the rear edge of the still, they enter the horizontal flue 7 and then the smokestack. This long path of the combustion gases is expedient, since it allows better utilization of their heat. Nevertheless, 40 percent of all the heat produced by the combustion of the fuel escapes through the smokestack. The total area of all the surfaces of the still that are bathed by the combustion gases, including the surfaces of the flues, make up the heating surface of the still.

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Since the temperature of the combustion gases is very high, especially at the beginning of their course, firebrick is used for the brick lining of the combustion gas passages and the first half of the flue, which are in direct contact with the flames.

In the still, beneath the flue, the steam coil 6 is placed. This consists of pipes with numerous openings through which steam enters the liquid, vigorously agitates it, and carries off the volatile components of the crude.

The still is usually charged with crude to 2/3 of its diameter. The surface formed by the crude at the level is termed surface of evaporation or mirror of evaporation. There is an outlet pipe 4 (or sometimes two of them), 250-300 millemeters in diameter, on top of the still, through which the mixture of steam and petroleum product vapors is removed. Since the process of boiling is violent, drops of crude are entrained with the petroleum-product vapors. To prevent this, transverse (discontinuous) baffles are set up in the outlet pipe. In passing around these baffles a number of times, the vapors are gradually freed from the drops of liquid. The still is provided with the crude intake pipe 1, with the crude discharge pipe 2, with the drain pipe 3, and the manhole 5, through which access to the still may be had for cleaning and repairs. It also has instruments for determining the temperature (thermometer) and the level of the liquid (by test-cocks).

In its principle of operation the batch still is reminiscent of the laboratory distillation flask. The crude is gradually heated to 100-130 degrees Centigrade, thus liberating the lightest fractions. Superheated steam is then led into the crude, and its temperature thus

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increased. When all the fractions desired have been evaporated from the crude, the burner is extinguished and the still cools, after which the mazut or residuum from the distillation of the crude is withdrawn from the still through the discharge line. The still is then recharged with crude oil and the process repeated.

The unit operates intermittently and is therefore called an intermittent plant, or batch plant.

Not only is the heating of the crude done in the still but also the processes of evaporation and separation of the vapors from the liquid. All these processes, however, are unsatisfactorily performed. For this reason the shell-still units were, as time passed, replaced by pipe-still units.

After leaving the outlet pipe, the petroleum-product vapors and steam (see Figure 61) take the subsequent route: dephlegmator 3, condenser 4, cooler 5, cut house 7. The dephlegmator is an iron cylinder with baffles which provide a large amount of cooling surface. Sometimes the dephlegmator consists of one pipe in another, with steam passing through the outer or ring space. The dephlegmator is designed to condense out of the vapors the highest boiling components of the crude, entrained by the volatile fractions. The liquid formed is called phlegma (from which the term dephlegmator) and flows back into the still.

Thus, the dephlegmator bears a crude resemblance to the rectification column. From the dephlegmator the vapors pass to the condenser (for condensation) and then to the cooler (for cooling). The cooled distillates are separated at the cut house into the separate

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distillates.

At the present time baton stills are employed almost nowhere.

46. The Continuously Operating Still Battery

The first continuous operating still battery was constructed at Baku in 1885; it consisted of 10 stills.

The layout of the still battery (Figure 63) is as follows: 15-20 stills are arranged in a row, each one being 18-20 centimeters higher than its lower neighbor. Thus, the last still is 2 to 3 meters lower than the first. On account of the slope so created the crude can flow by its own gravity from one still to the next. All the stills are joined by a single common brick lining and have a common horizontal flue and a common smokestack. The battery is fed by a common line. Its charging usually commences with the last still. After all the stills have been charged with crude to the required level, the burners are lit in succession under each of them. A slow heating of the crude to 120-130 degrees Centigrade then takes place, after which superheated steam is fed into the stills and the firing is increased. The light fractions are distilled from all the stills, and consequently the level in them falls. To maintain the necessary level in the stills, fresh crude is slowly fed into the first of them, whence it flows by its own gravity into the second, from the second into the third, and so on.

The temperature in the stills is gradually increased, and the rate at which the fractions are distilled also increases; therefore, in order to maintain a constant level in the stills the crude must be fed at an increasing rate. After the required temperature conditions

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have been established in all the stills, the rate of feed reaches its maximum (this rate being determined by the throughput of the unit). If we assume that the throughput of a battery is 1200 tons a day, the crude oil must be fed at the rate of 50 tons an hour.

Different fractions of the crude are distilled off from each still. In the first still the lightest fraction of the gasoline distillate is distilled, from the second a heavier fraction of that distillate, from the third one, a still heavier. Thus the crude becomes heavier and heavier as it flows from still to still and finally a mazut, with no more light-product content in it, flows out of the last still.

Steam is supplied to the different stills in amounts depending on the crude in them; the heavier the crude in any still, the more steam is supplied to it. The total consumption of steam reaches 10-20 percent of the crude. Gasoline vapors are distilled from the first 4 to 6 stills, kerosene vapors from the next 10-12, while the gas-oil-solar fractions are produced by the last stills.

Table 10 gives the quality of the distillates from the first 7 stills.

Comparison of the distillation results makes it clear how unsatisfactory the division of the distillates between the stills is. For instance, the distillate from the second still contains more light fractions in the 140-160 degree range than the distillate from the first still, and the difference between the distillates of the first five stills is very small.

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It is impossible to obtain commercial gasoline in a single operation from such still-battery operation, and therefore the gasoline-ligroin fractions are first distilled off, and these are then redistilled under still conditions.

TABLE 10

Still No	Gravity	Initial Boiling Point Degrees Centigrade	Percentage passing over up to temperature of Degrees Centigrade							End Point, Degrees Centigrade
			100	120	140	160	180	200	220	
1	0.762	79	5	28	56	78	90	95	98	220
2	0.767	92	1	28	59	80	90	94	--	223
5	0.777	105	--	20	52	76	88	94	--	227
7	0.792	123	--	--	21	52	75	87	--	247

The vapors of petroleum products emerging from the outlet pipe then pass successively to the dephlegmator, condenser, cooler, and cut house. Each still has a separate dephlegmator, condenser and cooler and operates as an independent part of the installation. This allows each of the stills to be taken off stream at any time for repairs or cleaning. Consequently, a still battery can operate for many months without interruption.

The separation of the distillates. In the cut house one distillate arrives from each still. The cut house has four receiving tanks: gasoline (the gasoline-ligroin fraction), kerosene, gas-oil, solar-oil, and carry-over.

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Only the ends of the pipes through which the distillates flow from the stills enter the cut house. Movable stand pipes are attached to the ends of these pipes, and may be placed in any of the tanks.

The simplest index that expresses the quality of the various distillates with a fair degree of accuracy is their specific gravity. The distillates are therefore separated on the basis of their specific gravity. Every hour the operator determines and fixes the specific gravity of all distillates. When the still battery is operating normally, the specific gravity of all distillates should be constant. If the specific gravity of the distillates increases, this means that distillation is too vigorous and that either the firing or the steam supply of the corresponding stills must be reduced. If this necessity has been established, then while the conditions of the still are being adjusted the distillate in question is flowed into the tank for the heavier distillates. If, on the contrary, the specific gravity of the distillates should fall, the firing of the stills must be increased or the feed of steam increased.

If a carry-over of crude has taken place in any still, the distillate from it is routed to the carry-over tank and measures immediately taken to adjust the regime in that still.

Thus, the distillates of various qualities are routed into the various receivers, for instance, the distillates of 8-10 stills with specific gravity from 0.780 to 0.845 are routed into the receiver for kerosene distillate, and thus on the average a standard kerosene distillate of specific gravity 0.826 to 0.828 is obtained.

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As has already been indicated, the quality of the distillates entering the receivers is checked every hour. If a distillate becomes lighter or heavier its routing must be changed. Assume that the kerosene distillate is composed of the distillates from the 8th to the 17th still. If the distillate becomes heavier, the fraction from the 17th still must be shifted to the gas-oil tank, and measures taken to adjust the conditions in the corresponding stills.

The throughput of still batteries may range from 500 to 3000 tons a day. According to the quality of the crude being processed, 20 percent to 50 percent of light distillates are produced.

Still batteries have many disadvantages. Let us indicate the principal ones.

(1) The very weak fractionating action of the stills, as a result of which the fractions overlap and the crude cannot be sharply separated into the corresponding distillates.

(2) The poor utilization of heat; the efficiency, i.e., the ratio between the useful heat acquired by the crude and the total heat liberated by the combustion of the fuel, does not exceed 0.5 to 0.6.

(3) The unwieldiness of the still installations.

(4) The fire hazard due to the simultaneous heating of substantial masses of crude.

(5) The possibility of the decomposition of the heavy hydrocarbons from the long stay of the crude in the heating zone.

(6) The high fuel consumption (both in the form of fuel directly burned in the burners and the indirect consumption, i.e., in the form of steam, which is supplied to the stills in large quantities).

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These disadvantages of still batteries forced a search for means of eliminating them or reducing their influence.

The creation of the so-called regenerative batteries, the construction of which began in 1908, represented a major achievement of the refining industry.

Such batteries were termed regenerative because the heat of both the exhausted combustion gases and the distillates and residue was regenerated or utilized in them.

The following must be reckoned among the achievements of the regenerative battery: the high percentage of heat utilized from the exhaust gases and residue, the preliminary vaporization of the gasoline-ligroin fraction which very greatly facilitates the work of the battery, and the provision of rectification columns. In the best regenerative batteries up to 45 percent of the escaping heat is returned for use, as a result of which the efficiency of the battery rises to 0.8, while the fuel consumption is cut in half by comparison with the old still batteries.

47. Vacuum-Still Installations

Lubricating oils were first produced in Russia at Gorkiy (in 1876) and Yaroslavl' (in 1879) and then at Baku.

Oils at first were produced only in batch-still installations. After the first continuously operating kerosene battery had appeared it was applied to the production of oils as well. The operation of the still battery for oils is in no way different in principle from that of the kerosene battery, but there are considerable differences in the equipment, owing to certain peculiarities in the distillation

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of masut to oils. The initial boiling point of the oil fractions is around 400 degrees Centigrade and the masut must be heated above that temperature to evaporate these fractions.

At such high temperatures the heavy molecules of the oil hydrocarbons break down into a number of smaller molecules. We recall that the boiling point of mazut can be lowered by the use of a vacuum and steam, which plays the part of a vacuum when mixed with hydrocarbons during distillation. As has already been explained, an insignificant quantity of steam is consumed with a high vacuum, while a large amount is used with a low vacuum.

Still batteries for oil are primitive units in which a high vacuum cannot be produced. In practice, residual pressures of less than 700 millimeters of mercury are not encountered in still installations. For this reason a large amount of steam (up to some tens of percent of the amount of the raw material) is consumed in the distillation of mazut to oil.

The Equipment of Still Batteries for Oil

The distillation of mazut is distinguished from that of crude by the high temperatures attained, by the employment of a vacuum, and by the high steam consumption. These peculiarities of oil distillation are reflected in the layout of the still and the head pipes. The remaining equipment of an oil battery is no different from that of a kerosene battery.

An oil still (Figure 64) differs from a kerosene still in having a considerably smaller capacity. Besides this feature, it has no flue, the firebox is removable, and the bottom is insulated by a layer of brick. The object of all these changes is to assure a smooth

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even heating of the masut and to prevent any possibility of overheating, since even a slight degree of overheating may lead to the rapid coking of the still. To assure greater firmness the floor of the still is made of boilerplate 18 millemeters thick. The flow of the combustion gases through the still is as follows: The firebox gases, passing under the floor of the still to its end at I, then rise and divide into two streams II, which bathe the still from both sides and then meet once more at the end of the still, passing out through the common horizontal flue III. The still is provided with steam collectors with a number of main pipes for the introduction of superheated steam, with pipes for admission and discharge of raw material and for withdrawal of the residue, as well as with a manhole for inspection and repairs.

2-3 head pipes are provided, which are used not only for reduction of the vapors but also for their partial separation into heavy and light fractions (Figure 65). This is achieved by not insulating the head pipes and putting two to three bends into them. The high-boiling hydrocarbons condense in the first bend, the lighter hydrocarbons in the second bend, and, finally, the most volatile hydrocarbons condense in the third bend. In this way three different fractions from a still are conducted from the three bends of the head pipe into the cut house.

The third bend of the head pipe ends in the dephlegmator 3 (see Figure 60), the structure of which is similar to the dephlegmator of the kerosene still. The condensation of the oil vapors is completed in the dephlegmator. The uncondensed vapors of decomposition products and the steam are sucked out by the exhauster 4, a device producing a vacuum in the still.

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The exhauster (Figure 66) consists of a cylinder inside which there are a number of conical nozzles. The water passes at first through the broad part of each nozzle and then through the narrow part. This produces a rarefaction; the vapors of the decomposition products, together with the steam, are sucked out and carried into the drainage system with the water. The exhauster produces a low vacuum, 60-80 millemeters of mercury (i.e., a pressure of 680-700 millemeters Hg absolute), which reduces the boiling point of the oil fractions by only a few degrees. The main purpose of the exhauster is not to create a vacuum but to quickly draw off the vapors of the oil fractions to avoid their overheating.

A few reconstructed oil batteries are equipped with head pipe still which increase the throughput of the batteries 20-30 percent.

48. Normal Operation, Starting and Closing Down of Still Batteries

The throughput of an installation, the separation of the distillates, and their properties, are mainly determined by the following conditions: (1) the temperature regime, (2) the quantity and temperature of the superheated steam, (3) the operation of the vacuum devides and (4) the condition of all the equipment and, in the first place, of the still itself.

Let us consider the operation, starting and shutdown of still batteries, and in particular, of oil batteries.

Temperature Conditions

This is very important for the operation of oil batteries. Heating of mazut above 380-390 degrees Centigrade causes its decomposition. The decomposition products entering the distillates affect

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their color, flash point and viscosity adversely; the color of an oil darkens, the flash point falls sometimes to 80-100 degrees Centigrade below the required standard, and the viscosity of the distillate is also reduced. Simultaneously with the decomposition of oil distillates a process of coke formation takes place, fouling the walls of the still. Such coke deposits put the still out of operation prematurely. For this reason, agitation in the still is done gently and smoothly, and the temperature in the last still does not rise above 360 degrees Centigrade.

Supply of Steam

A temperature of 360 degrees Centigrade is entirely inadequate to distill off all the lube oil fractions from mazut. To obtain the necessary separation of distillate in all the stills superheated steam is injected. The quantity of steam supplied increases from the first still to the last in connection with the increasing boiling point of the distillates. The steam consumption in the first still runs as high as 50 percent of the charge while in the last still it is 180 percent of the charge.

The superheat temperature of the steam is also of great importance. If insufficiently heated steam is supplied, the charge stock in the still will cool off. The steam is therefore customarily heated to 250-300 degrees Centigrade.

The vacuum should gradually increase from the first still to the last, with the difference between them usually amounting to 20-25 millemeters of mercury. The degree of vacuum is regulated by the delivery of water to the exhauster.

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Condition of the Equipment

If coking of a still commences, as may easily be detected by the appearance of dark spots, it should be taken off stream for cleaning and repairs. If defects appear in the head pipe, dephlegmator or exhauster, the still must also be shut down and the defect remedied.

Regulation of the Distillation Process

In a still battery this regulation is conducted on the basis of laboratory data and the readings of the control instruments. If any distillate becomes heavier, the firing of the corresponding still, or the delivery of steam to it, must be reduced. On the other hand, if the distillate becomes lighter, intensification of the firing of the still, or increase of the steam delivery, is required.

Starting an Oil Battery

The charging of the stills, and their agitation, commences with the last still which is charged with mazut and under which the burners are lighted. Then the charging and firing of the next to the last still proceeds. Thus, the whole battery, from the first to the last still, is gradually charged with mazut. By the time the charging of the first still is finished, the last one is already sufficiently heated, shortening the starting time of the battery. When 85-90 degrees Centigrade has been reached, the water is drained out of the stills. When 135-140 degrees is reached, superheated steam is led into the stills, the vacuum turned on, and water passed into the coolers and condensers. To compensate for a certain reduction in the amount of the raw material caused by the evaporation of the

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light fractions a small quantity of raw material is again charged (to maintain a constant level in all the stills). When the last still has reached 240-250 degrees Centigrade the residue is drained from it. The temperature in the last still gradually is brought up to the working temperature of 360-370 degrees Centigrade. By this time the steam consumption and the vacuum have been brought up to the working value (i.e., those established by the process chart).

During this period the production of the battery should also be brought up to normal.

The adjustment of the process proceeds in the following manner. The normal conditions are (approximately) determined, i.e., the definite temperatures, the definite amount of steam and the normal vacuum. Then this regime is made more precise and adjusted on the basis of the laboratory data, and the distillates are blended in such a way as to give distillates of the required qualities.

Cutting In A Still

This is done (after cleaning) in the operating battery in the following sequence: the still is filled with raw material, the bottom inspected, and if it is found to be in order the burners are lit. The exhauster is then turned on and water supplied to the coolers. When the temperature in the still has exceeded 150-160 degrees Centigrade, the delivery of steam is commenced. When the quality of the distillate nears that of the adjacent still, the still is included in the system and the normal operating conditions for it are established.

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The normal shutdown of a battery is accomplished by the gradual lowering of the fires in the combustion chamber and the reduction of steam delivery in such a manner that the fall of temperature in the stills does not exceed 25-30 degrees an hour. The distillates of all the stills during this period are routed to the carry-over tank. When the temperature of the liquid in the last still has reached 240-250 degrees Centigrade, all fires are extinguished, but the delivery of steam to the combustion chamber (through the burner) continues. The feed of fresh raw material and the discharge of residue from the last still is then stopped. Steam is passed for 2 to 3 hours through the liquid in the stills to cool it to 140-150 degrees Centigrade. At this temperature the vacuum may be turned off, and then the delivery of steam to the stills, and of water to the coolers, is stopped. The liquid from all the stills is drained through the coolers into the tank. After this, steam is blown through the stills for another three hours. Charge stock is pumped through the tar line to prevent the tar from solidifying.

Cutting Out A Still

If necessary one or more stills in need of cleaning or repair may be cut out of a system. The first stills require cleaning every 40 days, and the last stills every 20 days. To cut out a still the firing is gradually reduced until the walls blacken. The burners are then turned off, but the supply of steam continues to prevent sudden cooling of the still. The bypass gate valves are then opened, and the admission and discharge gate valves are closed. 3 to 4 hours after the still has been cut out, the flow of steam is reduced and

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the vacuum turned off. After the pressure in the still has been brought up to atmospheric level, the residue of the liquid is drained off through the cooler into the tank, and the still prepared for repairs.

49. Possible Accidents and Abnormalities in the Operation of a Still Battery

Interruption of the water supply leads to interruption of the condensation of steam and the products of decomposition in the exhauster. The vapors may thus spread through the plant and produce a fire hazard. For this reason the burners must be immediately extinguished and the steam flow to the stills reduced in case of any stoppage of the water supply.

Interruption of the steam supply will stop all the steam pumps and stop the agitation of the product in the stills, which may result in coking of the stills. To avoid this all burners must be immediately extinguished and the steam valves on all the apparatus closed, while the raw material flows by gravity (unless there are centrifugal pumps) into the battery. If the delivery of raw material is interrupted for a long time, the burners are put out, the steam supply to the stills reduced, and the battery shut down.

If a still ruptures and catches fire, it is cut out of the system and the fire-fighting service called.

If charge stock is carried over, it may enter the cut house and spoil the distillate.

Such carry-overs may be caused by:

(1) entrance of water into the still along with the steam, in

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which case the water must be immediately drawn off by the steam-blowing line;

(2) entrance of water along with the feed, in which case the firing in the first stills must be reduced and the water let out of them.

When a still is too full of charge stock, owing to obstruction in the runoff of the liquid into the next still, the gate valve to the raw material line bypassing the unadjusted still must be opened. Overfilling may also occur on account of stoppage of the tar pump or on account of solidification of tar in the cooler. In that case, the gate valve to the main line past the still is similarly opened, and the agitation in the still is reduced until the discharge of tar is in order again.

II. ATMOSPHERIC PIPE-STILL UNITS

50. The Advantage of Pipe-Still Units

In spite of various improvements and modifications made in still batteries, it has been impossible to eliminate a number of major disadvantages of this type of equipment: unwieldiness, great fire hazard and the possibility of breaking down the heavy hydrocarbons. Specialists have therefore worked on the problem of replacing the basic apparatus of the batch-still unit -- the shell still -- by another type of heating apparatus. The pipe-still heater was such an apparatus, and the first one in the USSR was constructed at Baku in 1925.

In contrast to the still batteries, the different operations in a pipe-still unit are conducted in different pieces of equipment:

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Heating in the Pipe still, evaporation in the evaporator or the evaporation section of the column, rectification in the rectification column, and so on.

The distillation of crude is conducted as a rule in two stages; in the first stage the light petroleum products are topped in atmospheric installations. These are gasoline, ligroin, kerosene and gas oil. In the second stage, in vacuum installations, oil distillates are distilled from mazut, i.e., the residue from the topping of crude.

Atmospheric plants may be single-flash, double-flash or multiple-flash. The oldest and most unimproved plants use the system of multiple flash, for instance, the Pintsch and Borman Plants, where each fraction has a separate column. These plants, in spite of the presence of a number of columns, are distinguished by poor distillate separation, unwieldiness, inconvenience of servicing and high fuel consumption.

In comparison with shell-still units, pipe-still units have the following advantages:

(1) the single flash allows distillation to be performed at lower temperatures than in shell-still units, since the volatile hydrocarbons lower the boiling point of the heavy hydrocarbons;

(2) consumption of fuel and steam is considerably lower in pipe-still units;

(3) the crude remains a considerably shorter time in the heater -- only 5 to 10 minutes -- and therefore the decomposition of hydrocarbons there is insignificant;

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(4) the fire hazard is not so great, since the volume of crude in the pipe still is about 40 times less than in the shell stills, with the same heating surface;

(5) the unit operates at high throughput;

(6) sharp separation of the fractions in the rectification columns and a high yield of light products from the potential are assured;

(7) servicing is convenient and allows rapid changeover of an installation from one set of operating conditions to another;

(8) a pipe still occupies one half to one third the space required by a shell-still.

51. Single-Flash Pipe-Still Units

Let us discuss the work of the pipe-still units that are most widely employed in the USSR.

A. Unit with radiation-convection type pipe still with horizontal flow of gases (Badger system). The heater (see Figure 32, 10) has the following dimensions: height, 5.4 meters; length, 12.7 meters; width, 8 meters; volume, 288 cubic meters. The convection chamber of the heater has the following dimensions: length, 4.1 meters, width, 4.9 meters, height (in the highest part), 2.7 meters and in the lowest part, 0.5 meters. There are 70 tubes in the radiation chamber, of which 47 are in the ceiling bank (two rows), and 33 in the sidewall banks (in single rows). All 70 tubes are connected by return bends to a single common pipe coil. There are 71 tubes in the convection chamber arranged in a single row and also connected with a single pipe coil.

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The outside diameter of all the tubes is 146 millemeters, but their inside diameter varies; that of the radiant tubes is 125 millemeters and that of the convection tubes is 131 millemeters; consequently, the walls of the radiant tubes are 10.5 millemeters thick, while the walls of the convection tubes are 7.5 millemeters thick. The radiant tubes must have thicker walls because they operate under more severe conditions than do the convection tubes. The total tube surface of the heater is 369 square meters, of which 56 percent is radiant-tube surface and 44 percent convection-tube surface. The efficiency of the heater is 70, the oil velocity through the tubes is 1.4 meter per second, the crude remains 10 minutes in the heater, and the charge of crude in the tubes is about 8 tons.

The rectification column has the following technical characteristics: diameter, 3 meters; height, 22.2 meters; 18 plates in the rectifying section and 5 in the distilling section.

The topping column consists of three independent columns which are superimposed: the ligroin, kerosene and gas oil columns. Each of these columns is connected by two lines to the rectification column, one of them bringing distillate from the rectification column, the other returning vapors to that column. The steaming column has 12 plates in all: 5 in the ligroin, 4 in the kerosene, and 3 in the gas-oil section.

The flow process (Figure 68) involves a single-flash pipe-still unit operating in the following manner:

The crude is drawn from the reservoir by a charging pump, by which it is pumped at a pressure of 15-16 atmospheres through two

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kerosene-spray heat-exchangers, three oil heat exchangers (the oil distillates arrive from another, vacuum installation), and one partial heat exchanger, in which the preheating is done by the solar oil vapors from the vacuum installation.

After being heated to 160-180 degrees Centigrade, the crude enters the sedimentation settler, where it is freed from water and sediment. The pressure after the exchangers falls to 10-12 atmospheres, i.e., 5-6 atmospheres is expended in overcoming the resistance in the exchangers. The crude is then pumped at 10-12 atmospheres through the heater, first through the convection chamber, then through the sidewall bank, the ceiling bank and the second sidewall bank. The crude leaves the heater at a temperature of 320-330 degrees Centigrade under 1 atmosphere pressure. From the heater the crude goes for evaporation to the 5th plate of the stripping section of a rectification column. The vapors of light petroleum products, together with steam, pass through the plates of the rectifying section of the column, while the mazut flows off by the four plates of the stripping section. The lightest product of the distillation of crude, gasoline distillate, together with the steam, enters the mixture condenser through the top of the column, then passes to the water separators and finally to the tank. Part of the gasoline distillate is returned to the column for reflux, so as to hold the temperature on emergence from the column at the level of 103-105 degrees Centigrade. Ligroin distillate at 135-145 degrees Centigrade is collected from the 19th and 20th plate; kerosene distillate at 215-225 degrees Centigrade is taken from three plates (12, 13 and 14). Solar oil is taken from the 7th and 8th plates.

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All these distillates pass through their respective stripping columns, in which the volatile fractions are evaporated off and returned to the column; the gasoline fraction is separated from the ligroin distillate, the ligroin fraction is separated from the kerosene distillate, and the kerosene fraction is separated from the solar oil. The ligroin and kerosene distillates and the solar oil, freed from the light fractions, flow by their own gravity from the bottom of the steaming columns into the corresponding coolers and then into the receivers. The hot mazut is collected by a pump at the bottom of the column and is routed for further distillation to the vacuum plant.

B. Single-flash plant with radiant-convection type heater with vertical flow of gases (Foster system). The throughput of these plants (Figure 69) ranges from several hundred tons to 2,000 tons a day. A plant of this type with a capacity of 500 tons of crude a day is described below.

The basic equipment of this plant consists of a series of apparatus described below.

The radiant-convection type heater with vertical flow of gases (see Figure 30) is 9.45 meters long, 4.8 meters wide and 7.74 meters high.

The radiant chamber of the heater has 30 tubes, 127 millemeters in diameter, arranged in two rows and connected with a single-pipe coil. The convection chamber has 42 tubes 100 millemeters in diameter, which are also connected to a single-pipe coil. The total surface of all the tubes of the heater is 291.8 square meters, of which 30

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percent is the surface of the radiant section and 70 percent that of the convection section. It must be noted that the surface of the hollow convection tubes is only 48.3 square meters, but thanks to the imposition of cast-iron rings on them, their surface is increased to 204 square meters, i.e., almost 4.2 times. Thus, the ring packing creates a larger heat-transfer surface. The rings also act to reduce the cross section of the convection chamber, which also improves the heat transfer by increasing the velocity of passage of the combustion gases.

A drawback of the ring packing is that it forms a peculiar kind of filter which holds back the soot and ash, increasing the resistance to the passage of the gases.

The efficiency of the heater is 72. The crude flows through the tubes at 1.25 cubic meters per second.

The rectification column is 22.8 meters high and 2.5 meters in diameter. It has 34 plates (of which 4 are exhausting) and inside separation sections forligroin, kerosene and solar distillates.

The flow sheet is shown in Figure 70. Crude oil is pumped under a pressure of 8-9 atmospheres through a gasoline-vapor heat exchanger, and then through the water and sediment separator 2. The crude so separated is pumped through the gas-oil and mazut heat exchangers 3 and heated to 150-180 degrees Centigrade. It is then preheated in the pipe still 4 to 300-315 degrees Centigrade after which it enters the rectification column 5 between the 4th and 5th plates. 4 plates serve for rectification of the residue, and 30 for that of the vapors.

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The vapors of the gasoline distillate mixed with steam emerge from the top of the column. The ligroin, kerosene and solar distillates, in addition to their main plates, also have stripping sections in the column for the final stripping of the light fractions they contain. The ligroin, kerosene and solar distillates, thus freed of the volatile fractions, flow off their respective plates in liquid state into the coolers 6, and then into the receivers.

52. Double-Flash Pipe-Still Units

A. Double-flash unit with preliminary evaporation -- the Soviet pipe still.

The Soviet pipestill units were constructed in 1935-1936 by Soviet specialists from domestically produced materials, and are among the very best and highest throughput crude-distillation plants. Their throughput runs up to 4500 tons of crude a day.

The basic equipment of the Soviet pipe-still unit consists of the following apparatus:

Two Nersesov system heaters are characterized by markedly developed radiant surface, much in excess of the convection surface. The radiant surface consists of two-row ceiling and side banks. Air is delivered to the heater after preheating in a recuperator (Figures 71 and 72).

The column is 28.98 meters high and 4.5 meters in diameter. It has 31 plates, of which 6 are in the exhausting section. Black solar is taken from the 9th plate, Diesel fuel from the 11th to 13th, kerosene distillate from the 17th to 19th, ligroin distillate from the 24th to 26th.

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The stripping column has 3 sections, each with 6 plates; they are placed in a single assembly above the evaporator. The preliminary evaporator consists of a hollow welded cylinder 4.8 meters high and 4 meters in diameter.

The Baku Worker heat exchangers are installed vertically. There are 6 for the circulating reflux, 6 for the kerosene distillate, 2 for Diesel fuel and 18 for mazut.

The blow sheet (Figure 73). Crude under pressure of 15-16 atmospheres is pumped through 24 heat exchangers (six are for circulating spray 1, six are for kerosene 2, two for Diesel fuel and ten for mazut 3) where it is heated to 118-120 degrees Centigrade. At this temperature, under the pressure of 5-6 atmospheres, the crude is settled in the water and sediment separators 4, after which it still passes through other mazut heat exchangers, and, heated to 160-180 degrees Centigrade, enters the preliminary evaporator 5.

The vapors from this evaporator are conducted by a heat pipe to the rectification column 6, while the crude, freed of gasoline, is pumped by two pumps through both heaters 10, in which it is heated to 330-335 degrees Centigrade. Then it unites into a single stream and enters the column. From the bottom of the column the mazut, freed from its light fractions, is pumped through the mazut heat-exchangers and the coolerinto its receiver. From the top of the column the gasoline vapors are taken off and conducted through the condenser, cooler and water-separator to its receiver. Part of the gasoline is returned to the column for refluxing.

Ligroin is taken off the 24th to 26th plate, its light fractions driven off in the stripping column 7, and after cooling it is routed into its receiver.

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The kerosene distillate is taken off the 17th to 19th plates, stripped in the stripping column, and pumped through heat exchangers into its receiver. An intermediate reflux is collected from the 22nd plate, pumped through the heat exchangers of the circulating reflux 1, and returned to the column at the 23rd plate. 35 percent of all heat used is obtained from the circulating reflux.

Diesel fuel (solar oil) is collected from the 11th to 13th plates, routed to the stripping tower and pumped through the heat exchangers. Black solar is collected from the 9th plate, which is routed to the container after cooling in the cooler.

The following is achieved by pre-evaporation: the load on the heater is reduced, since the crude is partly freed of the light fractions; the crude is completely water-free when it reaches the column, resulting in very sharp separation.

Soviet pipe-still units are among the most efficient of all plants: the recovery of gasoline is 99 percent of the potential yield, and the total recovery of all the light products together runs as high as 97-98 percent. The spread between the end point of gasoline and the initial boiling point of ligroin amounts to 3 to 5 degrees Centigrade, 5 degrees Centigrade between the end point of ligroin and the initial boiling point of kerosene, and 5 to 10 degrees Centigrade between the end point of kerosene and the initial boiling point of solar oil.

B. Double flash pipe still unit, with a radiant-convection type of heater with horizontal flow of the gases.

This unit has two heaters differing only in their dimensions

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and in the presence of two-row sidewall tube banks from that described in the preceding Subsection A of Section 51. (19 tubes are arranged on each side of the heater).

The crude flows through the heater in two streams. One of them flows through the 6 [sic] tubes of the lower row of the convection chamber, the side and ceiling tubes of the right half of the heater; the other stream flows through the 67 tubes of the upper row of the convection chamber, the side and ceiling tubes of the left half of the heater.

The aggregate heating surface of both heaters is 728 square meters, of which 51 percent consists of the radiant part and 49 percent of the convection part. The efficiency of the heater is 70 percent.

Columns: the plant includes two rectification columns and two topping towers of the following dimensions:

TABLE 11

	First Column	Second Column	Kerosene Stripping Tower	Solar Oil-Stripping Tower
Diameter, meters	3.03	3.33	1.81	1.50
Height, meters	17.63	14.94	5.72	4.17
Number of plates in the rectifying section	10	14	---	---
Number of plates in the stripping section	12	4	6	4
Spacing between the plates	0.61	0.61	0.61	0.61

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218 caps of the type shown in Figure 44 are placed on each plate of the first column, and 248 caps on each plate of the second column. In the first tower the steam consumption is 0.85 percent of the crude; in the second it is 0.64 percent, considerably less than in plants of other systems.

Heat-exchange equipment; the plant has 7 heat exchangers in all -- 1 ligroin, 1 kerosene, 1 solar oil and 4 mazut.

Figure 74 is a flow diagram. The crude is pumped from the reservoir by a centrifugal pump into the ligroin heat exchanger, where it is heated to 90 degrees Centigrade by the vapors of the ligroin distillates, together with steam. It then passes through the kerosene and solar heat exchangers 2, is heated to 115 degrees Centigrade and then goes to the water and sediment separator 3. After being freed from water and sediment, the crude then undergoes further heating to 170 degrees Centigrade in the mazut heat exchangers and is fed to the 12th plate of the first rectification column 4. This column has 10 plates in the rectifying section and 12 in its stripping section. The larger number of plates in the stripping section is necessary for stripping all the gasoline out of the semi-mazut. For this purpose steam is introduced into the column from the bottom. Since the heat of the preheated crude is insufficient to vaporize the whole of the gasoline fraction from it, part of the hot semi-mazut is pumped onto the third plate of the first rectification column. The gasoline vapors, mixed with steam, enter the condenser 5, the submerged cooler and the water separator 6. The gasoline distillate flows into its receiver from the water-separator.

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The semi-mazut is collected from the bottom of the column by a centrifugal pump and is pumped, as described above, in two streams through the two heaters 7, operating in parallel. The semi-mazut, heated to 330-340 degrees Centigrade, passes (in a single stream) to the 4th plate of the second rectification column 8. The ligroin vapors and steam pass from the top of the second column through the exchanger 1, and after giving up part of their heat in the preheating of the crude, are finally cooled in the cooler.

The kerosene distillate and solar oil in the liquid state flow into the stripping towers for evaporation of their light fractions. They are then routed through exchangers and coolers to their receivers.

Before entering its tank the mazut gives up part of its heat in the four mazut heat exchangers 2, through which it is pumped by centrifugal pumps.

The plant operates with good indices. The columns provide a sharp separation of the naphtha fractions, with a recovery of over 100 percent of the potential yield; in other words, the commercial columns operate better than the laboratory rectification column. The spread between the end point of the gasoline and the initial boiling point of the ligroin is no more than 10 degrees Centigrade. The plant exhibits a high degree of operational flexibility and can be readily changed over from one set of operating conditions to another.

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53. The Normal Operation of Pipe-Still Units

Each oil refinery has instructions for production, worked out in detail, together with a flow chart -- two documents with which all attendants must be well acquainted and which must be precisely fulfilled.

The instructions state how the work of the plant, and of each apparatus, is to be regulated; what measures must be taken in case of production difficulties or accidents; how normal shutdowns and emergency shutdowns are to be made; how the plant is to be started up after repairs, etc. Besides instructions of a general technological nature, instructions are also issued on safety measures and fire prevention. Failure to carry out any of the rules set forth in the instructions may lead to disruption of the normal operation of the plant and even to accidents.

Safety Measures

In our country an immense amount of attention is paid to safety measures. Not a single undertaking may be started without the authorization of the labor inspection and the organs of industrial hygiene and technical surveillance.

The following basic rules of safety must be observed in oil refineries:

1. All apparatus must be equipped with control instruments and with accident prevention devices.

2. Hazardous factors in the operation of the equipment must be eliminated by means of systematic inspection and repair.

3. The attendants must be well acquainted with the instructions for handling the unit, with the safety instructions and with

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the fire prevention instructions.

If the safety rules are disregarded at oil refineries, poisoning may occur by hydrogen sulfide, gases or gasoline vapors, gas or hot crude may take fire, personnel may be injured by electric shocks, and so on.

The most dangerous substance that can be liberated at an oil refinery is hydrogen sulfide gas, which is a powerful poison with an unpleasant smell of rotten eggs. In small concentrations it produces nausea, vomiting and headache. In high concentrations (of the order of 0.1 to 0.2 percent it causes death.

Petroleum gases and gasoline vapors are harmless in small concentrations, but may produce headache, dizziness and poisoning in higher concentrations.

The accumulation of gases and gasoline vapors involves great fire hazard, and in certain concentrations (from 4.9 percent to 15.4 percent for gases and from 1.5 percent to 6 percent for gasoline vapors) what is known as an explosive mixture is formed, which explodes with tremendous force at the slightest spark. To avoid accumulation of gases, the proper functioning of gas lines and valves must be carefully and continuously checked, together with the tightness of unions and couplings and the satisfactory operation of the ventilation system.

Fires may be caused by hot gas or by crude oil and vapor. For instance, the burner under a gas-fired boiler went out and gas accumulated in the firebox. The boilerman did not blow out the gas

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in the firebox but applied a light at once. An explosion followed, and the boilerman was burned on his hands and face.

To prevent such incidents the firebox and smoke flues must first be purged with steam before lighting the burner, in order to remove any possible accumulation of gas. Only after this has been done may a light be applied to the burner, and the gas cock must not be opened all at once but gradually.

In case of burns the affected part must be oiled with vaseline but not bandaged.

It must be emphasized once again that the principal means of preventing accidents is for the personnel to know well, and to fulfill undeviatingly and precisely, the rules of plant operation, of safety and of fire prevention.

The technological chart shows the limits within which the temperatures and pressures in the principal pieces of equipment in the installation must be held, as well as the revolutions per minute of the pumps, the levels in the columns, and the qualities of the products obtained. The regime of an installation must conform exactly to the technological chart confirmed for the installation in question.

Table 12 gives a few indices of the technological charts of two installations.

On regulating the regime of a unit, its production should remain constant, which is determined by the number of strokes per minute of the charge pump and by hourly gauging the feed in the feed-gauging tank. In some plants the rate of feed is automatically

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regulated by a meter connected with the charge pump.

Let us consider some of the most important factors in the conduct of the technological regime of a unit.

TABLE 12

	Double-flash unit	Soviet Pipe Still
Temperature, degrees Centigrade		
Entering the heater	---	105
Leaving the heater	340-345	330-335
Top of the column	105-110	105-110
Top of second column	146	---
Ligroin fraction	---	150
Kerosene fraction	245	200-220
Gas-oil fraction	315	270-275
Mazut	320	---
Mazut discharged	90	110
Gasoline in cooler	---	40
Pressure in column, in atmospheres:		1.2
In pre-evaporator	---	1
Saturated steam, minimum pressure	6	6
At the discharge of the mazut pump	6-7	15
At the discharge of the charging pump	6-7	15
In the dehydrator	---	5-10
Of the water	1.5	1.5

Operation of the Pipe Still

According to the purpose of the process (atmospheric or vacuum distillation, or cracking process), and also according to the quality

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of the feed, a definite temperature regime is established for each heater.

The temperature regime of a heater depends on a number of conditions. For instance, the inlet temperature of the charge depends on how the exchangers in the plant are functioning; the outlet temperature of the charge depends on the quality of the charge and on the prescribed depth of the recovery. This temperature is higher in vacuum heaters than in atmospheric and is still higher in cracking heaters than in vacuum. The temperature of the combustion gases passing out of the horizontal flues depends on the temperature of the charge stock entering the pipe still, and is always 150-160 degrees Centigrade higher than the latter. The temperature of the combustion gases above the partition wall depends on the purpose of the process, on the extent to which the radiant tube surface is developed and on the presence or absence of recuperation.

The principal factor in the operation of pipe stills is the regulation of combustion to assure minimum fuel consumption and prevent coking and burning of the tubes.

The normal process of combustion in the pipe still depends on the quality and quantity of the fuel. The best results may be achieved if the fuel is a low-viscosity fuel, readily atomized in the burner and free of water and sediment. (Gaseous fuel must not contain gasoline condensate).

Normal combustion also depends on the quantity of air fed to the pipe still. If the air is insufficient, combustion will be incomplete, resulting in overconsumption of fuel. Excess air will mix

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the combustion gases with cold air and cool them. Part of the heat will thus be unrationally lost and overconsumption will again result.

The course of the combustion process may be judged from the color of the flue gases leaving the smokestack: black smoke indicates incomplete combustion due to inadequate air; completely colorless smoke indicates that the combustion is proceeding with a great excess of air. Normal combustion produces light-gray smoke. When the pipe still is operating normally, the combustion gases should not contain less than 12 percent CO_2. The presence of CO indicates incomplete combustion.

The delivery of air to the pipe still is regulated by the aid of a blower. The combustion process is directly dependent, in addition, on the following factors:

The amount of steam delivered to the burner to atomize the fuel (when the burner is operating properly the color of the flame should be straw yellow; if the flame is smoky, the steam must be increased; if it is red, the steam must be checked);

The normal delivery of recirculated gases (if the recirculator stops running, the temperature of the gases above the partition wall immediately rises, which may result in burning the tubes);

The draft in the smokepipe (insufficient draft leads to incomplete fuel combustion and impairs the operation of the pipe still; the draft is regulated by the aid of a damper slide).

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The temperature of the charge stock on leaving the pipe still is very important for the normal operation of the unit and must be such as to assure the evaporation of all the light distillates; it usually ranges between 290 and 320 degrees Centigrade. Changes in the temperature of the charge stock affect the normal operation of the rectifying column, since such changes also change the quantity of the fractions evaporated, impair rectification and lower the recovery of light products from the potential yield.

The following causes act on the temperature of the charge stock leaving the pipe still:

Entrance of water and sediment into the fuel, thus disturbing the process of combustion and reducing the temperature of the emerging charge stock (in this case the water must be quickly drained out of the fuel tank and the operation of the burners regulated, and the fuel tanks must be carefully cleaned of water to prevent water from entering the fuel);

Large excess of air fed to the firebox (as a result of the heating of this excess air, the crude receives less heat and its temperature on leaving the pipe still will be lowered accordingly; in this case the blower must be turned down);

Damage to the regulator of the charge-stock flow (the amount of crude fed to the pipe still is reduced, thus increasing its temperature on leaving the pipe still; in this case the crude must be admitted through the water line);

Disrepair of the charging pump (resulting in reduction of the amount of crude delivered to the unit; in this case the emergency pump

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must be cut in).

The temperature of the combustion gases over the partition wall of the pipe still must be within a range of 750 and 850 degrees Centigrade, depending on the type of unit. Too high a temperature leads to burning of the tubes, while too low a temperature leads to a reduction of the unit's output since the amount of heat received by the crude in the convection chamber also changes in this case. If the temperature of the combustion gases over the partition wall rises, the flow of the fuel to the burners must be examined, and the blower opened more widely to intensify the flow of air into the firebox.

The pressure of the charging pump on discharge. When water mixed with the charge gets into the pipe still, the pressure at the pump rises sharply. To reduce the pressure, the water must be quickly drained from the dehydrator and the charging stock tank. Sometimes when a considerable amount of water has gotten into the charge, the output of the unit may temporarily have to be reduced.

The reason for the increase of pressure may prove to be the gradual fouling of the exchangers. It must be determined which exchanger is fouled, and that exchanger must be taken off stream for cleaning.

Temperature conditions in an atmospheric column. The prescribed quantity of the fractions is evaporated from the evaporator section of a tower if it is operating normally. Since evaporation absorbs heat, the crude is cooled 20 or 30 degrees Centigrade. As the vapors ascend in the tower, their temperature gradually falls,

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and on the top it will run around 110-120 degrees Centigrade. In a tower with 30 plates the average temperature drop per plate will be about (260-110): 30 = 5 degrees Centigrade. This average tempera--ture drop per plate is usually about 4 to 7 degrees Centigrade.

The temperature of the side-cut distillates will run about:

for ligroin	135-155 degrees Centigrade
for kerosene	195-230 degrees Centigrade
for gas oil	260-270 degrees Centigrade

The temperature of the mazut residuum is 260-280 degrees Centigrade, and will always be lower (by 20-30 degrees Centigrade) than that of the crude in the evaporation space. Steam is run into the tower for stripping the light fractions that still remain in the mazut.

The temperature at the top of the column determines the quality of the lightest product of all, gasoline. Fluctuation in this tempera-ture leads either to incomplete recovery of the gasoline, if it drops, or to production of a nonstandard heavy gasoline if it rises. This temperature may fall if water gets into the spray together with the naphtha. The latent heat of evaporation of water is 7 times as great as that of gasoline, and for this reason even a small amount of water has an appreciable effect on the temperature at the top of the column. To prevent water from entering the column, it is necessary to make sure that no water gets into the reflux tanks.

Fluctuation in the temperature at the top of the column may also result from stoppage of the spray pump, from sharp change in the steam delivery to the column, or from change in the temperature of the charge stock.

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Temperature of the superheated steam supplied to the column. The column receives a supplementary amount of heat from the delivery of superheated steam. When the temperature of this steam varies, the quantity of the tops also varies (given a constant quantity of steam). Therefore, if the superheat temperature of the steam should fall, its quantity must be increased, while, on the other hand, if this temperature should rise, the delivery of steam must be reduced.

Pressure in the column. The pressure in the evaporator section of an atmospheric column is always somewhat above atmospheric pressure, but it falls as the vapors ascend in the column. This fall in pressure is termed loss of head, and amounts to 2-6 millemeters of mercury for each plate of an atmospheric column, or a total of 40-60 millimeters of mercury, depending on the number of plates.

The level of liquid in the column and its sections must be held constant. Variation in this level affects the quality of the products. The level in the column is controlled by means of level regulators, or manually by means of test cocks.

The delivery of water for cooling the distillates. Water supplied in excess amount will cool the distillates well, but this method is too expensive and uneconomic. Delivery of water in inadequate amount is inadmissible, since there would be vigorous evaporation in the hot products arriving in the tanks, resulting not only in loss of the products but also in fire hazard. Besides this, high water temperature causes formation of scale in the tubes which is hard to remove.

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Therefore, the water must be supplied in such a quantity that the temperature of the products arriving in the receivers corresponds to the temperature fixed in the production chart; light gasoline is cooled to temperatures no higher than 35 degrees Centigrade, ligroin to not over 60 degrees Centigrade, and kerosene to not over 70 degrees Centigrade.

54. Laboratory Control of the Operation of Units

The above methods of regulating the operation of a unit with respect to temperature conditions, pressures, levels of liquid in the column, amount of steam and water supplied, cannot be chosen until the characteristics of the products are known. The regulation of the conditions of a unit at given output must be based on the characteristics of the distillates.

For instance, the temperature of the top of the column is set on the basis of a prescribed set of properties for the gasoline. If the gasoline produced is heavier than the prescribed value, the reflex must be increased. This lowers the temperature at the top of the column and the gasoline will become lighter, and be produced in lesser amount. If the gasoline is lighter than the value prescribed, the reflex is then checked somewhat. This will raise the temperature at the top of the column, and the gasoline will emerge heavier and in greater quantity.

The properties of the ligroin are regulated as follows: If it is too heavy, the gate valve on the ligroin line from the tower must be partially closed so that the flow of the ligroin from the tower will be less, while a larger amount of ligroin flows down along

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the plates, i.e., increases the ligroin reflex. Increasing the reflex lowers the temperature in the ligroin section, and the distillate will emerge more volatile and in lesser amount. On the other hand, when the ligroin distillate becomes too heavy, the gate valve on the outlet line from the tower is opened wider, so that more distillate will leave the tower, and the quantity of the ligroin reflex will be reduced. This leads to a temperature rise in the ligroin section and consequently to the distillate becoming heavier.

The properties of the kerosene and gas-oil distillate are regulated in exactly the same way by opening and closing the slides.

The residuum must not contain kerosene fractions. If it does, the steam supply to the bottom of the column must be increased to take off the kerosene fractions. The absence of kerosene fractions in mazut is characterized by a high flash point (175-185 degrees Centigrade) and a high initial boiling point, which should be 15-20 degrees Centigrade above the end point of kerosene.

These examples show how important laboratory control of the products is for properly regulating conditions in a unit. The laboratory takes samples of the products and charge stock every hour from a unit, and informs the unit of the results of the analysis.

First of all, the properties of the charge stock must be known.

The laboratory examination of crude consists in determining the properties of the crude itself, its potential content of distillates and their properties, and its content of harmful admixtures (water, sediment, salts).

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Knowing the properties and composition of the crude, the best refining method can be decided upon and the products and properties of products produced from it can be determined.

In speaking of the potential content of light petroleum products, this term means that according to the laboratory data the crude contains such and such a maximum quantity and such and such definite distillates of prescribed properties.

This is determined by the method of batch distillation of crude in a Gadaskin column or a TBP (true boiling point) apparatus (Figure 75).

Gasoline and ligroin content is determined as follows on distillation in a Gadaskin column. A few kilograms of crude are charged into the still and heated. A wide range gasoline fraction is taken off, running from the initial boiling point up to 125 degrees Centigrade, and a few narrow five-degree fractions from 125 to 150 degrees Centigrade. A second wide-range ligroin fraction from 150 to 175 degrees Centigrade is then taken, followed by a number of ten-degree fractions. The fractions obtained are then combined, i.e., mixtures or combinations are prepared.

Assume that we have been ordered to produce an aviation gasoline to contain 30 percent of the fraction up to 100 degrees Centigrade on determination with the standard still. We prepare the following combinations:

(1) the wide-range fraction plus the first five-degree fraction (from 125 to 130 degrees Centigrade);

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(2) the wide-range fraction plus two five-degree fractions (from 125 to 135 degrees Centigrade);

(3) the wide-range fraction plus three five-degree fractions (from 125 to 140 degrees Centigrade).

Analysis yielded the following results:

TABLE 13

	Distillation to 100 degrees Centigrade	Yield in percent
Wide-range fraction, from IBP to 125 degrees Centigrade	40	9
First combination, from IBP to 130 degrees Centigrade	37	10.5
Second combination, from IBP to 135 degrees Centigrade	34	12.5
Third combination, from IBP to 140 degrees Centigrade	30	14.5

The third combination, representing the fraction from the IBP up to 140 degrees Centigrade, proves to be the standard aviation gasoline, with a yield of 14.5 percent. This means that the potential content of aviation gasoline with this property is 14.5 percent for the crude in question. The potential ligroin content is determined by the preparation of combinations of the remaining five-degree fractions from 140-150 degrees Centigrade, of the wide-range fraction 150-175 degrees Centigrade, and of the ten-degree fractions taken above 175 degrees Centigrade.

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Assume that a mixture of the second wide-range fraction and four ten-degree fractions yields a standard ligroin, and that 7.2 percent of this standard ligroin is contained in the crude.

To determine the potential content of kerosene, the petroleum residue after distillation of the naphtha fractions is distilled in vacuum in a Gadaskin column under a pressure of 100 millimeters absolute. The following fractions are taken: a wide-range, up to 210 degrees Centigrade, 210-220 degrees Centigrade, 220-230 degrees Centigrade, 230-240 degrees Centigrade. Combinations are prepared and their properties determined in the same way as for gasoline.

Assume that the potential content of kerosene is 23.1 percent. Then the potential content of the aggregate of the light fractions in this crude will be 14.5 + 7.2 + 23.1 = 44.8 percent.

Distillation in a TBP apparatus gives a sharper fractionation than distillation in a Gadaskin column. Three-degree fractions are taken off the former apparatus, and a true boiling point curve (TBC) is plotted.

It is very important to know a crude's potential content of distillates. The system of premium payments to the attendants of units is based on recovery, in percentage of the potential, which characterizes their work and also the operation of the unit itself.

The properties of distillates obtained from the same crude may be roughly judged from their specific gravities, since the specific gravity of a distillate increases with its boiling point.

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The following other analyses are also made in the laboratory: determination of the fractional composition of gasoline, ligroin and kerosene, the initial boiling point of gas oil and masut, the flash point of kerosene, gas oil and masut. The technical conditions in an operating unit may be easily regulated according to these constants.

To illustrate, we give the regulation of the operating conditions in a unit according to laboratory data (data of Engineer Blagovidov). One of the units is distilling a crude containing 5 percent gasoline, 11 percent ligroin, 25 percent kerosene, 8 percent gas oil and 49 percent light oils.

The orders are to take off products with the following properties:

TABLE 14

	Initial Boiling Point, Degrees Centigrade	Fractional Composition to Degrees Centigrade					End Point in Degrees Centigrade	Recovery in percent of Potential
		86	100	150	200	270		
Gasoline	---	10	38	--	--	--	165	96
Ligroin	---	--	--	12	90	--	230	90
Kerosene	Not less than 200	--	--	--	--	80	300	96[1]

[1] total of light oils

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TABLE 15

	Initial Boiling Point, Degrees Centigrade	Fractional Composition to Degrees Centigrade					End Point in Degrees Centigrade	Recovery in percent of Potential
		86	100	150	200	270		
Gasoline	65	9.5	37	--	--	--	140	93
Ligroin	143	--	--	18	95	--	205	87
Kerosene	191	--	--	--	--	78	306	92[1]
Gas oil	310	--	--	--	--	--	--	--
Mazut	302[2]	--	--	--	--	--	--	--

[1] Total of light oils

[2] Of flash point 163 degrees Centigrade

Thus, the recovery of light oils was 50 percent, but their recovery from the potential did not hold up: 93 percent of the gasoline was recovered instead of 96 percent; ligroin, 87 percent instead of 90 percent; total light oils, 92 percent instead of 96 percent.

The following points may be noted from analysis of the characteristics of the products:

(a) The composition of the gasoline is too volatile: IBP, 140 degrees Centigrade instead of 165 degrees Centigrade, while 37 percent boils to 100 degrees Centigrade instead of 38 percent. The gasoline distillate is therefore, as they say, overlapped, which means that some of it has gotten into the ligroin distillate.

(b) The ligroin distillate is also too volatile: to 150 degrees Centigrade, 18 percent boils instead of 12 percent; to 200 degrees Centigrade, 95 percent boils instead of 90 percent, and the end point is 205 degrees Centigrade instead of 230 degrees Centigrade.

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Only 87 percent of the potential is recovered, instead of 90 percent, since part of the heavy ends of the ligroin have gotten into the kerosene distillate. Consequently, the ligroin, like the kerosene distillate, is overlapped.

(c) The kerosene distillate is overlapped among the fractions and at the same time its composition is heavier, since only 78 percent of the fraction boils up to 270 degrees Centigrade instead of 80 percent.

(d) The initial boiling point of the gas oil is 310 degrees Centigrade. Consequently, it does not contain kerosene fractions.

(e) The initial boiling point of the mazut is 309 degrees Centigrade; its flash point determined by Brenken tester is 163 degrees Centigrade instead of the normal 175 degrees Centigrade. Consequently, part of the kerosene has been included in the mazut.

Thus, under the improper temperature conditions of Table 16 the products obtained are either nonstandard (gasoline, kerosene) or too volatile (ligroin), resulting in lower recoveries from the potential than those prescribed in the assignment.

The outlet temperature of the crude from the first series of exchangers (see Table 16) is 180 degrees C, and drops to 130 degrees Centigrade out of the second series. Consequently, the stream of crude is being incorrectly routed through the exchangers and the first series of exchangers is operating at an underload.

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TABLE 16

	Outlet temperature of crude from the exchangers, degrees Centigrade		Feed temperature of crude to pipe still, degrees C	Outlet temperature of crude from pipe still, degrees Centigrade	Flue-gas temperature degrees Centigrade	Feed temperature to column
	I series	II series				
Unadjusted, incorrect conditions	180	130	140	310	750	280
Adjusted conditions	155	155	155	330	800	300

We use the gate valves to regulate the streams of crude in such a way that more passes through the first series of exchangers and less passes through the second. Then the outlet temperature of the crude from the first series of exchangers drops to 155 degrees Centigrade, while that from the second series rises to 155 degrees Centigrade. As a result of this regulation, the stream of oil will enter the pipe still at 155 instead of 140 degrees Centigrade in the first instance. Consequently, the oil temperature is increased 15 degrees Centigrade merely by having the exchangers operate normally.

As a result of the measures taken the outlet temperature from the pipe still is increased to 330 degrees Centigrade and that of the flue gases over the partition wall to 800 degrees Centigrade. The higher pipe-still outlet temperature results in a higher column-charging temperature (by 20 degrees Centigrade). The increased temperature of the crude oil naturally leads to intensified vaporization of the heavy ends and to making all the light oils heavier.

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The steam supply at the bottom of the column is increased to vaporize the kerosene fractions out of the mazut. To prevent the light oils from being made heavier thereby, the reflux at the top of the tower is increased, thus bringing down the temperature there 2-3 degrees Centigrade.

To make the kerosene distillate more volatile, the kerosene spray is intensified by partly closing the gate valve on the kerosene outlet line. At the same time, the supply of steam to the kerosene section is increasing for stripping the ligroin fractions.

To make the ligroin distillate heavier, we open the gate valve on the ligroin outlet line so as to reduce the reflux. Steam is added simultaneously in the ligroin stripping tower to strip the gasoline fractions. 2-3 hours after these operations, analysis of the distillates gives the following results:

TABLE 17

	Initial Boiling Point Degrees Centigrade	Fractional Composition to Degrees Centigrade					End Point Degrees Centigrade	Recovery in Percent of Potential
		86	100	150	200	270		
Gasoline	47	10.2	40.8	--	--	--	143	98
Ligroin	145	--	--	14	92	--	218	91
Kerosene	222	--	--	--	--	81	298	95[1]
Gas oil	312	--	--	--	--	--	--	--
Mazut[2]	321	--	--	--	--	--	--	--

[1] Total recovery

[2] (Br = 175 degrees Centigrade)

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The recovery of light oils from the crude still amounts to the same 50 percent, but all the products are standard and the extraction of gasoline and ligroin is good. The total recovery of light oils is now 95 percent (instead of 90 percent) on account of the incomplete recovery of the kerosene distillate. Since the pipe still cannot be forced further, steam is added in the column leading to an insignificant increase in the heaviness of the kerosene distillate and increase in its recovery.

55. Normal Shutting Down of an Atmospheric Pipe-Still Unit

Shutting down may be planned (normal) and unexpected (emergency).

During the operation of a unit the apparatus gradually clogs up, the pipe-still tubes become covered with coke and the individual parts of the apparatus erode. As a result the condition of the unit deteriorates: productivity is lowered and heat transfer in the pipe stills and heat exchangers is impaired, as is rectification in the column.

A large incrustation of coke in the pipe-still tubes is especially dangerous and intolerable.

Coke is gradually formed as the result of the incomplete splitting of the heavy fractions of crude during temperature fluctuations and interruptions in the flow of crude through the pipe-still. It accumulates on the tube walls and greatly impairs heat transfer. As a consequence of this impairment of heat transfer the crude receives less heat. Therefore, in order to maintain the pipe-still outlet temperature it is necessary either to reduce the throughput of the unit or to boost the work of the pipe-still so that the crude

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will receive the necessary heat regardless of the impairment of heat transfer. However, the latter measure may lead to superheating and hot spots in the tube; holes may form in it through which the crude will be ejected into the pipe-still and burned.

A considerable accumulation of coke in the tube can be approximately determined quite easily, since dark patches appear on the walls in these areas which are visible through the red-hot tube walls.

The accumulation of sediment in heat exchangers also impairs heat transfer and it is necessary to clean them from time to time. Condensers, coolers and settlers also clog up.

So that the unit may be operated under normal conditions it is periodically shut down for planned-preventive repairs. The working period of the pipe-still from the time it is put into operation until it is shut down for repairs is called its operating cycle. The normal operating cycles of atmospheric pipe-still units vary from 45 to 75 days depending on the condition of the apparatus and the quality of the distilled oil. The more contaminated the oil, the more frequently it is necessary to repair the unit. Before the pipe-still is shut down, its contents are switched over to a re-handling tank so as not to spoil the distillates in the tanks. First of all the throughput of the unit is lowered by slowing the pumping of crude. Simultaneously, firing is decreased so that the temperature of the discharged crude does not rise above the working temperature. When the throughput of the unit has been lowered 35-40 percent, the temperature of the oil at the pipe-still outlet is rapidly reduced to 240-250 degrees Centigrade. After this the burners are extinguished and the pump shut down, while the hot crude

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from the pipe-still is forced out into the column by steam. The coil pipe is drained of crude in 6-8 hours, i.e., at first the tubes of the convection section and then the radiant tubes. After the crude has been drained from the pipe-still the gate valve in the column is closed and steam blowing of the pipe-still tubes is begun, i.e., steam is admitted first in the radiant tubes and then in the convection tubes. Steam blowing is performed via an emergency line to the drainage system in 2 hours.

The return bends are opened after the coil pipe has been completely blown out. Hot return bends are opened easily, but it is difficult to open them if they have cooled. Therefore, the opening of return bends must not be delayed; it must begin immediately after the pipe-still tubes have been completely blown out. Only one plug is removed at first; this must be done very carefully in order to avoid scalding from steam or the remnants of hot oil which may still be in the pipe-still tubes. After several plugs have been removed all the return bends may be removed without danger. When all the plugs have been removed, the pipe-still is prepared for repair and cleaning. After the steam blowing from the pipe-still into the column has been completed, the liquid is pumped out of the latter and steaming is begun. Steaming lasts about 8 hours. If the column is to be welded, preparations are carried out according to definite specifications. The product is removed from the rest of the apparatus of the unit; it is opened and prepared for cleaning and repair.

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56. Production Difficulties and Possible Accidents in the Operation of Pipe-Still Units. Interruptions Caused by Accidents

Any disruption in the normal operation of a unit, which spoils production, lowers or interrupts the throughput of the unit is called an accident. Accidents can not rise spontaneously -- each one has a specific cause. Quite frequently the cause of accidents is a negligent attitude toward work or poorly trained attendants.

Besides, an emergency may develop from production difficulties caused by a decreased feeding of water, steam, electric power and crude.

A unit operates normally and without difficulty if attendants: are completely familiar with the layout of the unit, the condition of the apparatus and the physical and chemical properties of oil and oil products; have a conscientious attitude toward their work and precisely fulfill production specifications and regulations for accident and fire prevention; know how to orient themselves quickly to emergency situations and eliminate the causes.

It is possible to cite a number of examples when accidents and fires have occurred because of an unattentive attitude of attendants toward their work and their ignorance of the properties of crude and oil products. For example:

(a) to repair the gas apparatus a mechanic used an iron hammer, while according to the regulations a copper hammer was to be used in such cases. The air was saturated with gas. As a result of the impact of iron on iron a spark was generated which exploded the mixture of air and gas and started a fire in the unit;

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(b) while cutting out a heat exchanger, an operator closed the crude inlet and outlet valves; the feeding of hot masut was thus stopped for some time. The oil heated up and the pressure in the heat exchanger rose. The apparatus could not stand the increased pressure and exploded.

Let us now consider the most important production troubles and possible accidents, together with the measures for liquidating them.

Hot spots in a pipe-still tube.

A hot spot in a tube may be small if the jet of liquid flowing out of the opening is insignificant, and large if the jet of liquid issues violently from it.

To liquidate a small hot spot in a pipe still, the burners must be extinguished without stopping the supply of steam, and a normal shutdown made as described above.

To liquidate a large hot spot, all burners must be simultaneously extinguished, the charging pump stopped and steam admitted to the pipe-still. The liquid is then forced out of the tubes into the column without waiting for it to cool off. The further procedure is the same as for a normal shutdown.

Interruption or reduction of the steam supply immediately affects the normal operation of a unit, since most of the pumps in an oil refinery are driven by steam, the burners require steam for the normal combustion process, and steam fire-extinguishing apparatus is provided at all points where fire hazard exists.

If the steam supply at a unit is interrupted, the burners must be extinguished, the charging pump stopped, and steam drawn from the

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emergency line to blow the liquid out of the pipe still into the column. If the plant has no emergency charging line but has a centrifugal pump, this will be used to force the liquid out of the pipe-still into the column. In the absence of this possibility, it will be necessary to let new crude flow by gravity into the pipe-still under the pressure of the liquid in the charging tank, bypassing the heat exchangers and the dehydrator and forcing the hot liquid into the column. Simultaneously all doors in the pipe-still will be opened to cool it as quickly as possible. Until the steam appears again, all the gate valves on the steam line must be closed and the outlet cocks opened to free the steam line of condensate.

Interruption of the water supply to a unit also leads to undesirable consequences. If the interruption is only for a few minutes, operation may be continued on the emergency water tank, or on the supply of water in the tanks of the condensers and coolers, but if this is done the water outlet must be closed. In case of a long break in the water supply, the output of the pipe still must be reduced or operation even suspended entirely.

Insufficiency of water is first of all reflected in the operation of the gasoline condensers: the gasoline will be hot when it enters its receiver and will strongly evaporate, thus creating a fire hazard.

Interruption of the water supply is especially dangerous when mixture condensers are in operation at the unit, since in this case the condensation of the gasoline vapors will be stopped and the vapors expelled into the atmosphere.

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Interruption of electric power leads necessarily to the shutdown of units using a considerable number of centrifugal pumps. In other cases, for a short interruption (10-15 minutes) of electric power, the control and metering instruments and automatic mechanisms must be shut off, and the unit regulated manually and conditions observed by simple instruments, -- thermometers, manometers and testcocks. In case of prolonged interruption of electric power the pipe still must be shut down.

Interruption in the supply of crude. In case of a short interruption in the feed of crude (if the charging pump is out of order, the feed line in disrepair, etc) the unit is changed over to circulation. If the interruption is prolonged, the unit is closed down.

The entry of water into the crude is reflected in increased pressure on the charging pump and throughout all the equipment. In this case the burners are extinguished, the water cleaned out of the tanks, and normal operation again commenced only after this has been done.

If an exchanger becomes badly fouled or develops a leak, it is taken off stream for cleaning and repairs.

57. Corrosion of Equipment and Methods of Controlling It

Corrosion of equipment causes very great losses to the national economy.

Oil-refinery equipment is attacked mainly by naphthenic acids, hydrochloric acid, hydrogen sulfide and other sulfur compounds.

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Heat exchangers, pipe-still tubes, evaporators, columns and other equipment operating at high temperatures may be corroded by hydrogen sulfide. Partial condensers, submerged coolers, mixture condensers and dehydrators are corroded at the points of contact with water containing corrosive substances. Storage tanks are corroded by contact with hydrogen sulfide (roofs) and with water (at the bottoms). The outside parts of apparatus and piping is subject to rust.

Particularly strong corrosion takes place at plants refining crudes with large amounts of water, sediment and sulfur compounds (crudes of the Second Baku).

Methods of Controlling Corrosion

Petroleum must under all circumstances be dehydrated and desalted, and must not contain water or salts in quantities exceeding the prescribed norms.

The following measures are taken to reduce corrosion at oil refineries:

(a) Equipment, tubes and pumps are made of materials resistant to corrosion. The principal materials used for such equipment are cast iron, wrought iron, special alloys, alloy steels, i.e., steels containing nickel, chromium, aluminum, etc. For instance, the tubes in the radiant section of pipe-stills are made exclusively of alloy steel; pumps are made of cast iron, etc.

(b) Metallic protective coverings are used, i.e., the working surfaces of an apparatus are covered by a layer of metal that resists corrosion (chromium, tin, zinc).

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(c) Protective paints or lacquers are used, and the surfaces of apparatus or tubes covered with bitumen, cement, etc. This method is used only on apparatus operating at normal temperatures.

(d) Chemical reagents -- ammonia gas, lime, caustic soda -- are added to those parts of the apparatus where intensified corrosion takes place.

58. The Repair of Pipe-Still Units

The established operating cycle of a pipe-still unit should be strictly adhered to. A premature shutdown is undesirable since this adversely affects the production plan. Prolonging the run is also inadmissible since this leads to severe coking in the tubes and fouling of the equipment, which lengthens the time of repair many times over. A pipe-still must therefore be repaired precisely at the assigned date.

Repairs to units are current, intermediate and capital.

Current repairs are made every day during working hours. In this case apparatus that can be taken off stream temporarily without shutting down the whole process is repaired (for instance, exchangers, pumps, etc).

Intermediate repairs are basic in nature and are made at the end of every run. During the period of intermediate repairs, the tubes of pipe stills, coolers, condensers, exchangers and other apparatus are cleaned, defective tubes, return bends, bubble caps, gaskets, bolts on flanges and unions are replaced, and other work done on the repair and replacement of individual parts of the equipment. Intermediate repairs last 2-4 days.

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Capital repairs are made annually and last 10-30 days. During the period of capital repair, a large number of pipe-still tubes are replaced, the pipe-still walls relined, plates in the tower replaced, as well as towers themselves and the other major apparatus of the unit.

The repairs are executed pursuant to a special schedule termed defect schedule, covering all faults and defects that must be eliminated. The defect schedule is compiled during the course of an entire run, on the basis of the observed operation of the unit and on internal inspection of the equipment during the periods of shutdown.

Ordinarily a pipe-still is shut down at 2300 to 2400 hours so that it is ready for repairs the next morning.

As has already been stated, the cleaning of the tubes begins while they are still hot and the coke can be easily chipped off. The cleaning is done by special small air turbines (see Figures 101 and 102).

Safety rules during repairs. Accidents may occur when heavy bodies are shifted. To prevent them, all heavy bodies are moved by a special brigade of workmen, operating under the supervision of an experienced workman. All the winches, jacks, block and tackle, and other lifting mechanisms used in this work must be in good repair.

Cleaning and repair of apparatus is performed only after removal of the liquid, cooling of the walls to 50 degrees Centigrade, and careful purging or airing of the apparatus.

To avoid the entrance of liquid or gas from other apparatus into those being repaired all the pipe connections entering them must be capped.

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Especially careful preparation of the apparatus for welding work is necessary. For instance, welding work in a column may only be done after the product has been drained out, all piping entering the column has been capped, and steam blown through the column for 72 hours.

When welding is to be done in a pipe-still, steam is blown through it for 4 hours.

59. Starting Up a Pipe-Still After Repairs

The starting up of a pipe-still consists of the following operations:

(a) Inspecting the apparatus and testing it under pressure,

(b) cold circulation,

(c) hot circulation,

(d) bringing conditions in the unit up to operating levels.

After completion of repairs, a careful external inspection of the equipment is made. To make sure that the apparatus will operate normally, it must be tested under a pressure $1\frac{1}{2}$-2 times the working pressure. For instance, a pipe-still operating at 20 atmospheres is tested under 30. The testing pressure must be higher since the test is made at ordinary temperature, while the unit operates at high temperatures. To be sure that no leak will develop under working conditions it is tested at increased pressures.

Pipe-stills are pressure-tested in the following manner: The pipe-still is filled with charge stock, the outlet gate valve from pipe-still to column is closed, and the necessary pressure created in the system by operating the charging pump. After stopping the

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pump, all plugs in the pipe-still, together with the flanged unions and gate valves, are carefully inspected. If no leak is found anywhere, the pipe-still is considered ready for service. However, if a leak is found in the system, the charge is removed from the equipment, the defect is liquidated and the equipment again pressure tested.

Cold Circulation

After making sure that all the equipment of the unit is in order, we next proceed to the cold circulation. Cold circulation is necessary for testing all the equipment and pumps in operation.

Cold circulation is conducted in the following manner. The charge is collected by the pipe-still pump from the bottom of the column, then it is pumped through the exchangers, dehydrators and pipe-still and back to the column. In double-flash units the cold circulation is performed by two pumps: the charging pump and the pipe-still pump. The charging pump pumps the crude from the bottom of the first column (or from the pre-evaporator, wherever the crude is), through the pipe-still into the second column. The mazut pump from the bottom of the second column pumps the crude back through the exchangers and the mazut coolers into the first column or into the pre-evaporator. The cold circulation lasts 15-20 minutes. During this period all defects in the pipe-still, pumps and other equipment are disclosed. Hot circulation cannot be started before all defects noted have been remedied.

Hot Circulation

Before passing over to hot circulation, the coolers and condensers are filled with water, all product lines and overflow pipes

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are opened, and the control instruments turned on. The temperature is slowly increased to 105-110 degrees Centigrade at 10-15 degrees an hour, by one or two burners (turned low). At this temperature all the water remaining in the system after the washing out and pressure tests is evaporated. After this has been done, the temperature may be increased without danger by 20 to 25 degrees Centigrade an hour. Part of the light distillates are vaporized during this procedure, and the quantity of crude circulating in the system is reduced. Fresh crude is pumped into the column to hold the level in it constant. When the pipe-still outlet-temperature has reached 250-270 degrees Centigrade, the temperature at the top of the column reaches 80-90 degrees Centigrade. At these temperatures the hot circulation may be stopped and charging begun.

Change-over To Crude For the change-over to crude, on single-flash units, the gate valves on the charging line must be opened and crude supplied in the usual way, and the pumping of the mazut out into the tank must also be adjusted. In two-flash units, the charging pump is started up on the usual scheme: crude from the charging-stock tank is supplied through the exchangers to the first column or the pre-evaporator, while the mazut pump is switched over to pumping the mazut from the bottom of the second column through the exchanger system and coolers into the tank. The quantity of steam fed into the column is gradually increased. The distillates are routed through their overflow lines through the coolers and the receiving house into their receptacles. When enough gasoline distillate has accumulated in the reflux tank, the reflux pump is started to deliver the reflux to the top of the column. When the pipe-still outlet-temperature has reached 320-330 degrees Centigrade, the operating conditions may then be adjusted.

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Adjustment of Operating Conditions

It is only natural that when a unit changes over to crude, all the distillates should at first be nonstandard, since the operating conditions have not yet been adjusted, and the supply of steam, the reflux, etc have not yet been regulated. Let us take an example of how the operating conditions are adjusted.

Assume that the orders are to produce distillates of the characteristics given in Table 14. When temperatures near the operating temperatures have been reached, an approximately normal set of operating conditions is established: the pipe-still outlet temperature is brought to 300 degrees Centigrade, the temperature at the top of the column to 100 degrees Centigrade; then definite quantities of steam are delivered at various points of the column. After this the gate valves on the side draws are opened, as is usually done in the normal operation of a unit. After 3-4 hours, samples of the distillates are taken.

The laboratory analysis performed at the conclusion of these operations gives the results shown in Table 18.

The following characteristics are evident from the analysis:

(a) The composition of the gasoline is too volatile.

(b) The ligroin has a low percentage of evaporation up to 200 degrees Centigrade with a high end point, since some of the kerosene fractions passed into it; at the same time the increased yield of fractions boiling under 150 degrees Centigrade indicates that part of the gasoline fractions have also remained in the ligroin.

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(c) The kerosene distillate has a higher initial boiling point (220 instead of 200 degrees Centigrade); consequently, part of the light end of the kerosene (200 - 220 degrees Centigrade) has passed over with the ligroin distillate and made it heavier; the percentage of kerosene evaporated to 270 degrees Centigrade is reduced from 80 to 78.

(d) The gas oil and mazut contain 8 - 10 percent of the kerosene fraction.

TABLE 18

Distillates	Initial Boiling Point, Degrees Centigrade	Fractional Composition in Percent to Degrees Centigrade						End Point Degrees Centigrade
		86	100	150	200	270	315	
Gasoline	35	12	43	--	--	--	--	140
Ligroin	145	--	--	19	86	--	--	243
Kerosene	220	--	--	--	--	78	--	302
Gas Oil	290	--	--	--	--	--	--	--
Mazut	295	--	--	--	--	--	--	--

To improve the quality of the products it is necessary:

(a) to make the gas oil and mazut heavier by stripping them of their kerosene fractions; steam is passed into the bottom of the column for this purpose;

(b) to remove the kerosene fractions from the ligroin the delivery of the ligroin reflux is increased for this purpose, i.e., the gate valves on the ligroin side draw are partly closed;

(c) to lower the initial boiling point of the kerosene distillate, which is accomplished by the last-mentioned operation;

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(d) to make the gasoline distillate heavier; the reflux to the top of the column is reduced for this purpose.

TABLE 19

Distillates	Initial Boiling Point, Degrees Centigrade	Fractional Composition in Percent to Degrees Centigrade						End Point Degrees Centigrade
		86	100	150	200	270	315	
Gasoline	38	10.5	40.5	--	--	--	--	143
Ligroin	140	--	--	17	92	--	--	218
Kerosene	190	--	--	--	--	81	--	300
Gas Oil	300	--	--	--	--	--	8	--
Mazut	301	--	--	--	--	--	5	--

After operating 3 hours under the modified conditions, the characteristics of the products are tested (Table 19).

As a result of these modifications, the following characteristics are obtained:

(a) The ligroin distillate is still too volatile (17 percent to 150 degrees Centigrade); to make it heavier the steam feed to the ligroin section is increased.

(b) The increase in the ligroin reflux lead to a reduction in the initial boiling point of the kerosene distillate to 190 degrees Centigrade due to part of the ligroin fractions passing over in it; to strip that distillate of these fractions the steam supply to the kerosene section is increased.

(c) The gas oil and mazut still contain a certain amount of the kerosene fractions; to evaporate them the pipe-still outlet

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temperature of the crude is raised from 310 to 315 degrees Centigrade.

After 3 hours analysis of the products gave the following results:

TABLE 20

Distillates	Initial Boiling Point, Degrees Centigrade	Fractional Composition in Percent to Degrees Centigrade						End Point Degrees Centigrade
		86	100	150	200	270	315	
Gasoline	38	10.5	40.5	--	--	--	--	144
Ligroin	143	--	--	15	91	--	--	222
Kerosene	209	--	--	--	--	80	--	298
Gas Oil	305	--	--	--	--	--	5	--
Mazut	310	--	--	--	--	--	3	--

Since the ligroin distillate is still too volatile, the increase in the steam supply to the ligroin section is continued, after which the percentage of the ligroin fraction evaporated to 150 degrees Centigrade is reduced to 13, while the initial boiling point rises to 146 degrees Centigrade.

In this manner a set of conditions has been found that can be accepted as working conditions. The separation of cuts under these conditions is sharp, and no overlapping whatsoever of the fractions is observed.

60. Cutting In and Cutting Out Individual Pieces of Apparatus in the Operation of a Unit

During the operation of a unit the need for repairing dehydrators, exchangers and tubular coolers sometimes arises. These pieces

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are so installed that they may be cut out of operation.

To cut out an exchanger, the gate valves on the bypass lines (around the exchangers) are opened, then those on the admission and outlet lines are closed. One of the gate valves on the crude admission or outlet line is kept open two or three turns until the exchanger has cooled off completely, otherwise the crude on being heated might crack the exchanger. Exchangers are repaired and cleaned with the crude and product lines capped.

Exchangers are cut in as follows: Admission and outlet lines are gradually opened. Simultaneously the gate valves on the bypass line are gradually closed. If the exchanger has been cleaned with insufficient care, foreign articles from the repair work may remain in it, and therefore on closing the gate valves on the bypass line the pressure shown by the pump manometer must be checked. If this rises above normal, the exchanger must be cut out again and the cause for the rise in pressure found.

A dehydrator is cut out by opening the gate valves on the bypass line around it, and on the feed and discharge lines for the crude. The crude is then drained out of the dehydrator.

The following is the sequence of operations for cutting in a dehydrator settling tank:

The discharge line is closed, the air valve is opened and the gate valve of the crude feed line to the dehydrator is opened a single turn. After crude appears at the air valve it is closed, the crude admission and discharge lines are fully opened and the bypass line is closed.

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In doing this the temperature at the dehydrator inlet must be watched, together with the pressure at the charging pump. A dehydrator cannot be quickly cut in, since less crude will enter the pipe still during this time, and its temperature at the pipe-still outlet will consequently rise, which may lead to coking of the pipe-still tubes.

To cut out a cooler, the distillates are routed around it, after which the water is turned off.

A cooler is cut in in the reverse order.

III. DISTILLATION OF MAZUT IN PIPE-STILL UNITS

61. The Disadvantages of Shell-Still Oil Batteries and the Advantages of Pipe-Still Vacuum Units

Shell-still oil batterieshave the same disadvantages as shell-still kerosene batteries have. In addition to the general disadvantages common to kerosene batteries, oil batteries have others peculiar to them: the great shortage in the recovery of lubricant distillates, which may run as high as 25-30 percent, resulting from the decomposition of the mazut; the low quality of the distillates owing to the lack of rectification; the impossibility of getting high-viscosity distillates since high temperatures, which are not allowable in shell-still units, are necessary to vaporize them.

To eliminate these disadvantages pipe-still vacuum units were designed. The first such Soviet unit was built at Baku. It was a high-vacuum pipe still for distilling petroleum tar. Later (in 1930) vacuum units of other systems also appeared at Baku.

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In contrast to shell-still oil units, in which the temperature of the charge cannot be raised above 300-370 degrees Centigrade since if masut is heated in a shell still for 10-12 hours conditions favoring its decomposition arise, no decomposition of the distillates in a pipe-still unit takes place if the masut is brought up to 420-425 degrees Centigrade since the charge remains in the pipe still only a short time (not over an hour). Heating the charge to 425 degrees Centigrade, in combination with a vacuum and superheated steam, makes it possible to evaporate all the lubricant distillates out of the charge without decomposing them. In vacuum towers vacuums of the order of 680-700 millemeters (60-80 millemeters absolute) are established by vacuum-creating devices. Bubble-cap trays make it possible to secure a sharp separation of the various oil distillates. Besides this, the distillates here have a better fractional composition and color and higher flash points and viscosities than those obtained from shell-still batteries.

We shall now familiarize ourselves with the vacuum units most extensively used in the USSR.

62. Vacuum Pipe-Still Plants

A. Vacuum pipe-still units with radiant-convection type heaters having a vertical flow of gases (Alco systems).

The principal equipment of this unit is described below (see Figures 76 and 77).

The pipe-still radiant-convection type heater with a vertical flow of gases differs from the heaters of this type that have already been described only in that its convection chamber is somewhat shifted, and that the radiant tubes end at the partition wall and do not enter the convection chamber.

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The convection chamber has 96 tubes with a total surface of 271 square meters, while the radiant chamber has 68 tubes with a total surface of 252 square meters. The heater is 8 meters high, 6.1 meters wide and 8.84 meters long.

The vacuum tower of the plant 3 differs from other towers in the arrangement of the bubble caps (see Figure 45) and the structure of the lower or stripping section. The tower is 26 meters high, its diameter in the rectifying section is 5.18 meters and in the stripping section 2.29 meters. The stripping section of the tower is narrowed to assure high velocity of the tar passing through it. The tower has 24 plates in all, of which 4 are in the stripping section.

The stripping tower consists of 4 independent sections placed one on top of the other. Each is 3.75 meters high and 1.0 meters in diameter.

A vacuum is created by the action of the barometric condenser 4 and the steam jet 5. A vacuum is formed in the barometric condenser by the condensation of the steam and gas-oil vapors. The barometric condenser serves at the same time as a heat exchanger in which the charge stock is heated by the heat of the gas-oil vapors.

The four shell-and-tube exchangers 1 preheat the charge stock by using the heat of the distillates and tar being discharged.

The vacuum receptacles 6. The distillates leaving the tower cannot flow into receivers by their own gravity, since for this the external pressure would have to be overcome. For this reason they

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first enter hermetically closed containers with the same vacuum as in the tower. The distillates are then pumped from the vacuum receptacles into containers.

The flow chart (Figure 77). The mazut, heated to 50 degrees Centigrade, is pumped through the heat exchangers placed in the barometric condenser and the four distillate and tar heat exchangers 1. The mazut, heated to 200-230 degrees Centigrade is then pumped in two streams through the pipe still 2, where it is heated to 420-430 degrees Centigrade. To increase the oil velocity through the radiant section, superheated steam is admitted together with the mazut. In the evaporator section 3 of the tower, on account of the high temperature, the low pressure (140 millemeters absolute) and the admission of superheated steam, 70 percent of the lubricant distillates are evaporated. A considerable amount of heat is consumed in this evaporation. This heat is given up by the charging stock, which is thereby cooled from 425 to 400 degrees Centigrade. The residue after the evaporation of the distillates -- which is lubricating oil tar -- flows off through four stripping plates, and superheated steam is fed into the bottom of the tower to strip it of the lubricant distillates remaining in that tar.

The mixture of lubricating oil vapors ascends through the plates, from which the following distillates are taken off: gas oil from the 21st plate, spindle-oil distillate from the 17th, machine-oil distillate from the 13th, light-cylinder oil distillate from the 9th, and heavy-cylinder oil distillate from the 5th. All these distillates enter the exchangers and then the vacuum receptacles 6, from where they are pumped through the submerged coolers 7 into their receivers.

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The gas-oil vapors passing out of the top of the column together with steam are condensed in the barometric condenser, while the uncondensed products of decomposition and air are pumped out through two 10-inch pipes by the two-stage steam jet 5. A vacuum of 680 millemeters Hg (80 millemeters absolute) is established. In the evaporation space of the tower the absolute pressure is 140 millemeters. Consequently, 60 millemeters of pressure is lost in the tower in overcoming the resistance of the bubble caps.

B. Vacuum pipe-still units with radiant-convection type heaters with a horizontal flow of gases.

In this plant (Figure 78) crude is distilled to tar in two stages: crude to mazut in the atmospheric section and mazut to tar in the vacuum section.

Even though the flow chart of the unit differs only slightly from the flow chart described in the foregoing Subsection A, there are nevertheless substantial differences in equipment design.

The vacuum pipe still of the unit is analogous to the atmospheric pipe still described earlier, but it is somewhat smaller: the inside diameter of the tubes is 77 millemeters instead of 134 millemeters , and the aggregate tube surface is 210.5 square meters instead of 414.5 square meters in the atmospheric pipe still. The small diameter of the tubes is necessary for creating high oil velocities through the heater.

The vacuum tower (see Figure 40) has the following peculiarities: (1) it is low (only 8.7 meters) but wider than all existing vacuum towers, having a diameter of 6.4 meters; (2) it has only 8

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plates for taking off three or four cuts and no evaporator section at all.

There is a rack in the column under the mazut feed line. 1392 round bubble caps are arranged on each plate.

A fault of the tower is the absence of an evaporator section and the very rigid attachment of the plates to the barrel, of the tower as a result of which there are often cases of the edges of the plates breaking away from the walls. This produces cracks through which liquid drips, thus ruining the rectification. The evaporation space of the tower is very small, and this causes spashing of tar into the motor-and-tractor oil section.

The advantages of this tower lie in its simple design, the small loss of vacuum owing to the small number of plates, the high vacuum (40-45 millemeters absolute, while in the tower of the preceding unit it is 120-140 millemeters absolute).

The unit has two barometric condensers: a large one, 1.11 meters in diameter, and 2.08 meters high; and a small one, 0.2 meters in diameter and 0.465 millemeters high. The vacuum section has no exchangers, and the heat of the tar is used in the atmospheric section. This is also an advantage of the unit, since it has no additional unions through which air might be sucked in and impair the vacuum.

The flow chart (see Figure 78). Mazut heated to 250-260 degrees Centigrade is collected by the hot pump from the bottom of the atmospheric tower and is delivered first to the vacuum pipe still 1, where it is heated to 410-420 degrees Centigrade, from

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which it goes to the vacuum tower 2. In the evaporator section of the vacuum tower the temperature of the charging stock falls to 360-370 degrees Centigrade. The spindle, machine, motor, or cylinder oil distillates are taken from each two plates (the 6th, 4th and 2nd). However, in general distillates may also be taken from other plates, according to the characteristics that may be required. The distillates are taken by centrifugal pumps direct from the plates and are pumped through coolers into their containers.

From the top of the column the gas-oil vapors pass through the partial condenser 3, giving up a part of their heat to the crude, and then to the gas-oil condenser 4. The steam is condensed in the barometric condenser, while the products of decomposition are removed by the three-stage ejector 7.

The tar from the bottom of the tower is pumped through the exchangers of the atmospheric unit to the asphalt plant.

As will be noted from the description, the scheme of operation of the unit is very simple. This makes it very easy to regulate the operating conditions: any change in the system of conditions requires only 3 to 4 hours, while it takes 8 to 12 hours in other units.

C. The Soviet vacuum pipe stills. The first Soviet vacuum pipe stills were constructed at Baku and designed to distill the tars produced by the lubricating oil shell-still batteries. As we have already learned, when mazut is distilled in shell-still batteries the low vacuum makes it impossible to separate all the lubricant fractions from the mazut, and a considerable part of them still remains in the tar. To utilize these valuable fractions, the tar was

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passed through the high-vacuum Soviet pipe-still. This separated up to 45 percent of cylinder-oil distillates with a viscosity of 5 to 5.5 degrees Engler at 100 degrees Centigrade, and a residue of bitumen ready for use was obtained.

At the present time there is no further necessity for such an operation, since mazuts as a general rule are processed in pipe-still units; for this reason the high-vacuum pipe-still units are utilized for other purposes (for redistillation of lubricating oil distillates with soda).

Figure 80 gives a general view of the Soviet vacuum unit. On the basis of the experience with vacuum pipe-stills, Soviet specialists have projected and constructed new Soviet pipe-still units distinguished by high technological indices.

We present a characterization of the principal equipment of the new Soviet pipe-still plants.

The pipe still has a greatly developed radiant section with roof and side tubes arranged along the walls. Up to 77 percent of all the heat liberated in the heater is delivered in the radiant section to the product heated. This pipe-still is analogous to that shown on Figure 72.

The vacuum tower 6 is low but large in diameter; it has 10 plates, 7 of which are designed to rectify the vapors and 3 to rectify the residue. The tower has a constricted stripping section. The stripping section is separated from the rectifying section by a screen baffle to prevent tar from splashing on the plate. As will be seen from the flow chart, besides the upper reflux an

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intermediate reflux may also be used in the tower (from the 2nd-5th plates). A high vacuum is produced in the tower (50-70 millemeters absolute). The plates rest on corner irons so that they cannot separate from the sides; and their spacing has been increased to 900 millemeters (to facilitate repairs and cleaning).

The unit has five exchangers for distillates and tar.

The flow chart (Figure 79). The mazut heated in the exchangers is fed in two streams into the pipe still. Each stream passes through two rows of radiant and convection tubes, arranged at the bottom of the pipe still, and side tubes. The roof tubes are single-current, and therefore the two streams are combined into one before entering them. The charge enters the tower 6 heated to 420 degrees Centigrade. Spindle, transformer and gas-oil distillates, or machine-oil distillates, or machine-oil distillate and bright stock, may be taken from the tower. In the former case gas oil is taken from the top of the tower and semi-tar from the bottom, while in the latter case a wide-range fraction is taken from the top and tar from the bottom. The vacuum in the tower is produced by the barometric condenser 9 and the jets 10. The tar or semi-tar is pumped through two exchangers 4 into the container. The distillates are also pumped through exchangers 2 and 3.

63. <u>The Operation of Vacuum Pipe-Still Units and the Regulation of the Process of Distillation</u>

The normal operation of vacuum units is determined by temperature conditions, the degree of vacuum, the amount and temperature of the steam feed and the condition of the equipment. Since

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vacuum pipe-stills operate under more severe conditions, than atmospheric units, especially thorough inspection is necessary.

Temperature Conditions

The outlet temperature of the charge stock from the pipe-still is determined by the fraction that is to be produced from the mazut. Usually it does not exceed 420 degrees Centigrade, but it may be raised to 430 degrees Centigrade if superheated steam is supplied to increase the velocity of oil movement through the radiant tubes. It must be strongly emphasized that this temperature may be held only under the normal throughput of the unit. If the throughput should drop for any reason, the temperature must be immediately lowered or the pipe-still tubes will be coked. The outlet temperature of the charge stock is largely affected by the extent of the preheating of the mazut in the exchangers; as soon as the normal operation of the exchangers is impaired and the mazut begins to arrive less heated at the pipe still, the outlet-temperature from the pipe-still will fall.

As we have already remarked, the temperature of the charge stock in the evaporator space is always 25-30 degrees Centigrade below its outlet temperature from the pipe-still as a result of the heat lost in evaporating the distillates. The temperature at the top of the tower is established on the basis of the quality of the distillate to be separated and is regulated by the admission of gas-oil reflux.

The degree of vacuum exerts a decisive influence on the separation of lube oils. At high vacuums, each 10 millemeters of mercury

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fluctuation in the vacuum produces a change of 10-20 degrees Centigrade in the boiling point. Consequently, fluctuation in the vacuum will exert about the same influence on the distillation process as fluctuation in the pipe-still outlet temperature of the charge stock.

The flow of water into the barometric condenser is regulated in such a way that the gas-oil vapors and steam are completely condensed and the temperature of the water discharged from that condenser does not exceed 60-70 degrees Centigrade. The temperature of the water entering the condenser is very important, since the higher it is the higher the vapor pressure of the water will be and the poorer the vacuum. The degree of vacuum depends on the temperature of the water, as follows:

Temperature of water in degrees Centigrade	10	15	20	25	30
Vapor tension of water in millemeters of mercury	9.2	12.8	17.5	22.8	31.8

If the temperature of the incoming water is 30 degrees Centigrade, the residual pressure cannot be lower than 31.8 millemeters of mercury, since that is the value of the vapor pressure of water at this temperature.

The work of the steam jet depends on the steam pressure; the higher this is the higher the vacuum will be. For this reason it is very important to hold the steam pressure high and constant, since fluctuations in the steam pressure also affect the vacuum.

The quantity of steam admitted to the tower must be sufficient to evaporate all the lube oil fractions out of the tar. However,

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excess steam is also harmful, since it will impair the vacuum by its vapor pressure. The superheat temperature of the steam should be kept at the 400 degree Centigrade level.

Regulation of the Distillation Process

The process of distillation in a vacuum pipe-still unit is regulated according to the readings of the control instruments and the laboratory analyses of the characteristics of the distillates.

If the distillate is to be made heavier, the gate valve on the product take-off from the tower is opened wider; this will reduce the quantity of phlegma available for the reflux into the lower plate and more of the fractions will be vaporized.

To make the distillate lighter, the gate valve on the take-off is slightly closed.

Assume that the orders are to produce distillates of the following characteristics:

TABLE 21

	Viscosity		Brenken Flash Point	Freezing Point
	E_{50}	E_{100}	Degrees Centigrade	Degrees Centigrade
Spindle-oil distillate	219-3.2	--	170	- 15
Motor-oil distillate	--	1.8-2.3	205	0
Cylinder-oil distillate	--	4.6	250	- 5
Residue - mark III bitumen	--	--	--	--

The following operating conditions are established for purposes of orientation: temperature at top of column, 190 degrees Centigrade; pipe-still outlet temperature, 425 degrees Centigrade. The downcomers

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are opened and a definite quantity of steam is admitted into the tower.

Distillates of the following characteristics are obtained under these conditions:

TABLE 22

	Viscosity		Brenken Flash Point	Freezing Point
	E_{50}	E_{100}	Degrees Centigrade	Degrees Centigrade
Spindle-oil distillate	2.6	--	180	- 18
Motor-oil distillate	--	2	225	- 12
Cylinder-oil distillate	--	4	250	- 7
Bitumen Mark III	--	--	--	nonstandard

It will be seen from the analyses that the spindle-oil distillate produced is volatile, and the bitumen is nonstandard. To make the spindle-oil distillate heavier, the temperature at the tower top must be increased to 195 degrees Centigrade or the reflux reduced for the same purpose.

To make the bitumen heavier, the admission of steam to the bottom of the column is increased. In this case increasing the pipe-still outlet temperature is not recommended since it is high enough already.

Under the modified operating conditions, distillates of the following characteristics are now obtained: [Table 23 on next page.]

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TABLE 23

	Viscosity		Pranken Flash Point	Freezing Point
	E_{50}	E_{100}	Degrees Centigrade	Degrees Centigrade
Spindle-oil distillate	2.75	--	185	- 16
Motor-oil distillate	--	2.3	225	- 11
Cylinder-oil distillate	--	4.5	265	- 5
Bitumen Mark III	--	--	--	standard

As we see, the motor-oil and cylinder-oil distillates and the bitumen are now standard, but the spindle-oil distillate is still too light. For this reason the temperature at the top of the column is now increased to 200 degrees Centigrade, and the viscosity of the spindle-oil distillate rises to 2.95. The set of operating conditions thus found may be accepted as regular working conditions.

64. Factors Involved in Accidents in the Operation of Pipe-Still Units

Let us now consider the most frequently encountered accidents and trouble in the operation of vacuum units.

1. Interruption of the steam supply leads to an emergency shutdown, since all the steam pumps stop, the supply of process steam to the tower is interrupted, and the degree of vacuum falls on account of the cutting out of the steam jet.

2. Interruption of the water supply also leads to a shutdown, since the barometric condenser is shut off and pressure impairs the vacuum.

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3. Interruption or reduction in the charge stock feed may lead to rapid coking of the pipe-still tubes. In this case the unit must be shut down.

4. Impairment of the vacuum, adversely affecting the operation of the unit, may result from the following causes:

(a) insufficient quantity of steam supplied to the jet as a result of which the steam feed must be increased;

(b) drop in the steam pressure, making it necessary to increase the quantity of steam supplied and increase the steam pressure to normal;

(c) insufficient water supplied to the barometric condenser; in this case the water supply must be increased;

(d) excess water in the barometric condenser, flooding the condenser and disrupting its operation;

(e) an open cock at some point in the equipment through which air is drawn; this drawing in of air must be located and eliminated;

(f) leakage at some point of the equipment; if a leak is found in the unions it must be remedied, if it is in the body of any apparatus the unit must be shut down for repairs.

5. If pipe-still tubes are burned out, the same procedure as in atmospheric units is to be followed.

6. The tar does not come out, since the pumps "do not take it". This is due to formation of vapor lock in the admission line. The admission line must be blown clear by a pump into the tower and put in service again.

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7. The temperature at the top of the column fluctuates sharply and then falls. This may happen if water gets into the tower along with the reflux.

65. Starting Up and Shutting Down a Vacuum Pipe-Still Unit

After a unit has been repaired, it is pressure tested with masut using the charging pump or the tar pump. A rigorous pressure test of the vacuum tower with steam or air is particularly important, since the slightest leakage may lead to the aspiration of air and the impairment of the vacuum in the tower. In pressure testing a tower with air, all the unions must be smeared with soapsuds. This is done to discover leaks, since a soap bubble will appear at any point where there is the slightest leakage.

After the pressure test the unit is ready to operate. The pipe still, exchangers and tower are charged by the charging pump (up to the established level). Then to test the working of the pumps the liquid is circulated back and forth between the tower and the pipe still for 10 or 15 minutes, by the tar pump or charging pump. After this we proceed to the hot circulation along the same lines, gradually increasing the temperature in the system. The vacuum apparatus is started. When the pipe-still outlet temperature reaches 300 degrees Centigrade, the change-over to operation on the charge stock may be made. Superheated steam is run into the tower and the tar pump cut in to pump out the residue from the tower. The pipe-still outlet temperature is gradually increased and with it the throughput of the unit, and regulation of the distillation conditions may already be started.

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The operating cycle of a vacuum unit lasts for not over 70 days, after which it must be shut down to repair equipment and to clean the coke from the pipe-still tubes and the fouling and coke from the tower plates and exchangers.

A vacuum pipe still is shut down in the following manner. The shutdown begins by reducing the firing in the heater of the pipe still in such a way that the outlet temperature falls 20-25 degrees Centigrade an hour. At the same time the throughput of the unit is reduced (to 60-70 percent). The products are routed into the carry-over tank. When the pipe-still outlet temperature reaches 320 degrees Centigrade, all the burners are extinguished but the charging still continues until the outlet temperature has fallen to 260 degrees Centigrade. The product is then blown out by way of the passage from the pipestill to the tower. This operation takes 4 hours, after which the pipe still is blown out in the opposite direction into the drain system. The admission of steam to the tower and the refluxing at the top of the tower are continued until the temperature at the top of the column has fallen to 100 degrees Centigrade. The residue is then pumped out of the bottom of the column. After all these operations the vacuum is turned off, but the steam admission to the column continues until the vacuum has been completely broken. After the vacuum has been turned off, the upper manhole of the tower is removed and it is steam blown for another 12 hours.

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CHAPTER EIGHT

THE CRACKING PROCESS

I. Thermal Cracking

66. The Significance of the Cracking Process

The use of cracked gasoline is increasing with every passing year and it is assuming a larger and larger proportionate place in the total production of motor gasoline. In 1932 cracked gasoline accounted for 20 percent of the total production of motor gasoline, in 1937, 50 percent, and in 1946, over 70 percent.

How is this immense success of the cracking process explained? Why is cracked gasoline displacing straight-run motor gasoline?

The demand of aviation for aviation fuel is steadily growing, and all of the crude suitable for producing aviation gasoline is being used for this purpose. Only an insignificant amount of crude still remains for the production of straight-run motor gasoline. Consequently, it would be necessary to increase the production of crude to satisfy the demand for motor gasoline, which is also growing rapidly, that is, to make immense capital expenditures on drilling and construction. However, the reserves of crude are limited, and therefore each ton of crude produced must be used to the maximum possible advantage. These considerations were the reason why the cracking process, which makes it possible to produce gasoline from mazut, the residue from the distillation of crude, came into existence.

The cracking process is effectuated by a more complex pro-

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cedure and requires equipment different from that employed in the distillation of crude. It must be noted that the cracking process makes it possible not only to increase the quantity of the gasoline obtained from crude, but also to improve its antiknock properties.

67. The Chemistry of the Cracking Process

As we have already learned, when crude is heated in a closed vessel the light fractions are evaporated first, then the heavier fractions, while the mazut is concentrated in the residue. On further heating, the large molecules of mazut are broken down into the light molecules that make up gasoline and gas. This ability of the heavy hydrocarbon molecules (mazut, gas oil) to break down into lighter molecules under the action of high temperatures is taken advantage of in the cracking process. Light petroleum products are not the only ones formed in the course of the cracking process, products heavier than the cracking stock are formed as well, all the way to the solid substance, coke. Consequently, not only do reactions of decomposition take place in cracking but reactions of condensation as well.

Let us consider the behavior of the various classes of hydrocarbons in cracking.

Paraffin hydrocarbons: Under the action of high temperatures the paraffin hydrocarbon molecule splits into two parts, one of which is also a paraffin hydrocarbon. However, one has fewer carbon atoms while the other is an unsaturated hydrocarbon:

$CH_3 - CH_2 - CH_2 - CH_2 - CH_2 - CH_2 - CH_2 - CH_3 \longrightarrow$

normal octane, a liquid (contained in gasoline)

$CH_3 - CH_2 - CH_2 - CH_3 + CH_3 - CH_2 - CH = CH_3$

butane, a gas butylene, a gas

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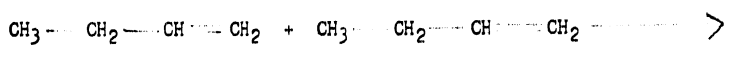
Unsaturated hydrocarbons can be split into two parts: one a saturated hydrocarbon with a small number of carbon atoms, and the other an unsaturated hydrocarbon with an additional double bond.

The polymerization reaction is particularly important for the unsaturated hydrocarbons, and consists of the condensation of two, three or more than three molecules into a single molecule. For instance, a molecule of octylene may be obtained from two molecules of butylene:



bytylene butylene

octylene, a liquid

The products of the condensation of unsaturated hydrocarbons are called polymers. Six-member naphthenes may be dehydrogenated and thus transformed into aromatic hydrocarbons (aromatization).

Aromatic hydrocarbons are very stable compounds and cannot be split into smaller molecules under the action of high temperatures, but on the contrary undergo the condensation reaction, thus forming a molecule with a larger number of carbon atoms and a molecule of hydrogen.

Coke is formed as the result of deep condensation of aromatic hydrocarbons.

The side chains may be split off from both aromatic and naphthenic hydrocarbons with formation of a cyclic hydrocarbon

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(for example, benzene is formed on the splitting off of a molecule of ethylene from ethylbenzene).

All the multiplicity of chemical transformations that take place during cracking may be reduced to the following reactions:

(1) to the reaction of cleavage of the molecule in two parts,

(2) to the reaction of splitting off hydrogen (dehydrogenation),

(3) to the reaction of splitting off the side chains from cyclic hydrocarbons,

(4) to the reaction of aromatisation,

(5) to the reaction of condensation.

68. The Principal Conditions Affecting the Cracking Process

The principal conditions that affect the course of the cracking process are the quality of the cracking stock, the temperature to which it is heated, the duration of the heating and the pressure.

Cracking Stocks

Experiments have shown that the course of the cracking process depends to a considerable extent on the fractional and chemical composition of the cracking stock. The heavier it is, the more readily it will be cracked. Given the same temperature conditions and heating time, more gasoline will be obtained from a heavier stock (mazut) than from a lighter stock (kerosene). The condensation reaction also proceeds more easily in cracking a heavy stock. Consequently, a larger amount of coke is formed in cracking mazut than in cracking gas oil or kerosene. For instance:

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TABLE 24

Cracking stock	Specific gravity	Percent gasoline	o/o carboids
Gas oil	0.833	29.7	0.01
Paraffinic mazut	0.906	31.4	5.07

To avoid rapid coking of the equipment, the formation of carboids (coke) is, in plant operating practice, permitted only in an amount not exceeding 0.1 percent. Under this condition the yield of gasoline from mazut does not exceed ten percent, while from gas oil it is 40 percent. Thus, even if a heavy stock does crack more easily, it does not necessarily follow that it is a better cracking stock. On the other hand, gas oil is the best cracking stock from the point of view of coke formation.

The chemical composition of the cracking stock is also of no little importance.

The paraffin hydrocarbons are the easiest of all to crack. Paraffinic cracking stock is also the best from the point of view of coke formation, since it produces the smallest amount of coke. A heavy aromatized cracking stock is the worst material for cracking, for its principal reaction is one of condensation, leading to formation of a large amount of coke.

The unsaturated and naphthene hydrocarbons occupy a position intermediate between the paraffin and aromatic hydrocarbons.

Temperature Conditions

The following law holds for chemical reactions: the velocity of chemical reactions doubles for every 10 degrees C rise in temperature. This law has been found to apply, with certain deviations,

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to the reactions that take place in the cracking process.

Let us take the reaction velocity of the cracking process as being determined by the percentage of gasoline formed in one minute when a high temperature acts on the cracking stock. Assume that 0.1 percent of gasoline is formed in 1 minute at 425 degrees C. If the temperature is raised 10 degrees to 435 degrees C, the reaction velocity will be doubled, i.e., 0.2 percent of gasoline will be formed. If the temperature is then raised another 10 degrees to 445 degrees C, then 0.4 percent of gasoline will be formed, that is, double the amount again, and so on.

The velocity of cracking may be increased not only by raising the temperature but also by increasing the heating time of the cracking stock, and doubling the heating period will also double the velocity of cracking.

As has already been stated, at 425 degrees C 0.1 percent of gasoline is formed in one minute, 0.2 percent after two minutes, 0.4 percent after 4 minutes, etc. Thus, by combining temperature and heating time, the most advantageous conditions, or, as they say, the optimum conditions, may be selected for cracking a stock in the commercial equipment.

For instance, 30 percent of gasoline may be obtained from mazut, using the following temperatures for a determinate time of heating:

At 400 degrees C, the heating time will be 12 hours
At 425 " " " " " " " 2 "
At 450 " " " " " " " 20 minutes
At 475 " " " " " " " 2 "
At 500 " " " " " " " 0.6 minute

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Thus, if the stock is heated for 12 hours at 400 degrees C, the same result will be obtained as on heating it for 0.6 minute at 500 degrees C. Pressure also plays an important role in the cracking process, and for this reason commercial plants as a rule employ high pressures, not infrequently running up to 40-70 atmospheres. In cracking under atmospheric pressure, the stock to a considerable extent is in the vapor phase. Very high temperatures are necessary for vapor-phase cracking, which on the one hand creates conditions favorable to coke depositions, and, on the other hand, requires equipment having a great volume.

The quantity of vapor is reduced under increased pressure; the mixture of vapors and liquid becomes more uniform, occupies less space, requires equipment of smaller size and is cracked at lower temperatures. As a result the deposition of coke is lessened.

Pressure also exerts an influence towards reducing the gas yield and on the chemical composition of the gasoline; in particular, the output of unsaturated hydrocarbons is reduced.

69. The Development of the Cracking Process

The first observations on the decomposition of the hydrocarbons were made during the past century, and in 1890 Engineer Shukhov was the first in the world to design a cracking plant. But since the demand for gasoline at that time was not great this method remained unutilized for a long time. Only under the Soviet regime (in 1923) was the first Shukhov-Kapelyushnikov cracking plant constructed at Baku.

The first industrial unit (Figure 81) was very simple and consisted of a still with a furnace under it. The liquid charged into the still was heated to 400-410 degrees C under a pressure of 10-15

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The products of decomposition passed into the condenser through the still-head tube. The unit was quickly clogged up by coke and could not operate for more than three days, after which the coke had to be cleaned out.

As time went on more complex plants appeared, with pipe stills to heat the cracking stock and reaction chambers for the cracking itself. (Figures 82 and 83). In the USSR an imported industrial cracking plant (Vickers system) was constructed in 1928. The plant proved very unsuccessful and operated only a short time, after which it was converted into a straight-run unit.

In 1930 the construction of a Winkler-Koch plant was completed. Although the use of plants of this system is widespread in the USSR, they have a number of faults which prevent high gasoline yields from being obtained. In 1930 the construction of a Winkler-Koch plant was completed. Although the use of plants of this system is widespread in the USSR, they have a number of faults which prevent high gasoline yields from being obtained. In 1934 Soviet engineers designed a plant that was free of the principal faults of the Winkler-Koch system. This plant received the designation "Nefteproyekt-system two-coil cracking plant". During the last ten years, all the cracking plants built in the USSR have been of this system exclusively.

Before the war a combined selective-cracking plant with a gasoline yield of about 50 percent was constructed at one of the refineries.

Finally, catalytic cracking is the latest achievement of technology, making it possible to obtain a high yield of high-grade gasoline.

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70. Various Forms of the Cracking Process

The cracking process may be conducted at different temperatures and pressures, and on different cracking stocks: light and heavy, in the presence of hydrogen, oxygen and various catalysts. A number of forms of cracking are distinguished, according to the various conditions employed.

Mild cracking is employed for cracking heavy petroleum products, especially mazut and tar, at temperatures of 450-460 degrees C and pressures of 25-40 atmospheres. More severe conditions cannot be used in this case on account of the heavy coking. Mild cracking is occasionally used to reduce the viscosity of heavy petroleum products for use as boiler fuel. This process is termed mild cracking of mazut.

Mild cracking is more often used not as an independent process, but in conjunction with deep cracking at one and the same unit, to obtain 5 to 10 percent of cracked gasoline from heavy cracking stock, together with the maximum amount of kerosene-gas-oil fraction, which is then used as stock for deep cracking.

The deep cracking process. The kerosene-gas-oil fractions obtained from the light cracking of mazut is used, together with fresh gas oil pumped into them, as stock for deep cracking. The temperature is held at the level of 500-510 degrees C, and the pressure at 45-60 atmospheres. The yield of gasoline is 30-35 percent the amount of the cracking stock. This is the system that is most widely used in the USSR (Nefteproyekt-system two-coil plants).

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The vapor-phase cracking process. The pressure is held at atmospheric level or slightly above it, and therefore all of the stock is present in the vapor phase, that is, in the form of vapor. This process requires a temperature of about 600 degrees C.

The vapor-phase process is not widely employed because of its disadvantages: the intensified coking and high yield of gas on account of the elevated temperature, the high fuel consumption (almost double that of a liquid-phase process) and the low productivity of the plant.

The vapor-phase process includes the pyrolysis of light stock (kerosene, gas oil), which is conducted in equipment specially adapted to this purpose at 650-700 degrees C.

The basic object of pyrolysis is to obtain aromatic hydrocarbons (benzol, toluol, xylol, etc) and gas.

Selective cracking. To crack a heavy stock relatively low temperatures are required, while they must be high to crack a light stock. In cracking mazut, which is a mixture of heavy and light fractions, a certain intermediate temperature is selected which is too high for the heavy fractions and too low for the light ones.

A cracking plant of this type consists of a number of furnaces; in each of them a close-cut fraction of the charge stock is cracked at the temperature most suitable for the fraction in question (optimum temperature). This method of cracking allows the yield of gasoline to be considerably increased.

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Catalytic cracking in recent years has come into widespread use. This is cracking in the presence of a catalyst. Catalytic crackers give high yields of aviation and motor gasolines and considerably less residue and gas than other types of cracking units. All the coke is deposited in the catalyst, from which it is burned out at regular intervals. Since there are a number of catalyst chambers in a plant, the process is continuous.

Hydrogenation (cracking in the presence of hydrogen) is effected at 450-500 degrees C and under very high pressures, up to 200 atmospheres and beyond. Since coke and heavy residues are formed in cracking owing to insufficiency of hydrogen, hydrogenation makes it possible to increase the yield of gasoline to 75-80 percent by adding hydrogen from outside.

The reforming process, so-called, is used to improve the characteristics of gasoline, ligroin and kerosene, and is effected at 510-550 degrees C and pressures of up to 70 atmospheres.

Oxidation cracking. An experimental pilot plant for oxidation cracking on the Dubrovaja system, i.e., cracking in the presence of oxygen, has been designed and constructed in the USSR.

The flow chart of this plant is as follows: kerosene or gas oil is heated in a pipe still to 400-420 degrees C, enters the reaction chamber (a brick-lined tank) in the form of vapor, and is mixed there with air. Owing to the high temperature the oxidation reaction takes place very rapidly and energetically, and the temperature of the mixture rises to 560-600 degrees C on account of the heat of reaction. The products of the reaction are then cooled and fractionated. This process gives considerable yields of high-grade aviation fuel and valuable oxidation products.

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71. The Characteristics of Cracked Products

Cracking produces the following products: gas, raw gasoline, which is termed pressure distillate, cracked fuel oil, coke and certain intermediates.

Gas is obtained in quantities of 7-10 percent in liquid-phase cracking, up to 20 percent in vapor-phase cracking and up to 50 percent in pyrolysis. Its composition varies according to the type of cracking; its unsaturated hydrocarbon content runs up to 30 percent for liquid-phase cracking, 45 percent for vapor phase cracking and between 30 and 35 percent for pyrolysis.

Gasolines. The gasoline yield also varies for the various types of cracking, amounting to 25-28 percent in Winkler-Koch units, 30-35 percent in two-coil units, up to 45 percent in cat crackers, and up to 80 percent in hydrogenation plants.

Cracked gasolines differ from straight-run gasolines, and also differ among themselves, in their content of unsaturated and aromatic hydrocarbons.

TABLE 25

Group Chemical Composition of Gasolines (in percent)

	Naphthenes	Aromatic Hydrocarbons	Unsaturated hydrocarbons	Paraffin hydrocarbons
Grosniy straight-run gasoline of end point 175 degrees C	30	6	--	64
Grozniy cracked gasoline	6.2	16.4	27.6	49.8
Vapor-phase gasoline	5-15	40-45	45-50	---
Hydrogenated gasoline	26	5	16	59

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As will be observed from the Table, cracked gasoline, in contrast to straight-run gasoline, contains unsaturated hydrocarbons: vapor-phase cracked gasoline up to 50 percent, liquid-phase up to 27 percent, hydrogenated up to 16 percent. It also contains aromatic hydrocarbons: vapor-phase gasoline has 40-45 percent of these and liquid-phase has up to 16 percent. Cracked gasolines therefore have high octane numbers: liquid-phase gasolines, 65-66; vapor-phase, 70-76.

A great fault of cracked gasolines is their tendency to form gum, owing to their content of unsaturated hydrocarbons.

Cracked fuel oil is a heavy viscous product (of about 80 degrees Engler), of black color, with specific gravity 0.97 to 1.00 and higher. It is used as furnace oil.

72. A Winkler-Koch Cracking Plant Is a Liquid-phase Unit with Two Furances

Below is a description of the principal equipment of such a plant.

1. The small furnace or low-pressure furnace (Figure 84) serves to take off the kerosene and gas oil fractions from the feed stock and for the mild cracking of the mazut. It is 7.3 meters long, 5.7 meters high, and 5.9 meters wide. Its coil consists of 174 seamless tubes, of which 53 are roof tubes and 120 are in the convection chamber.

2. The large furnace or high-pressure furnace is designed for the deep cracking of the kerosene and gas oil fractions produced in the unit and of fresh gas oil pumped into be added to the cracking stock. Its design and dimensions are analogous to the deep cracking

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furnace of the Nefteproyekt-system cracking plant. Its coil (Figure 85) consists of 230 tubes (70 in the roof screen, 64 in the first preheater, 16 in the second preheater and 80 in the reaction section). It is 10.1 meters long, 8.1 meters wide, 7.1 meters high.

3. The evaporator (Figure 86) is a vertical hollow apparatus 15.6 meters high, and 2.74 meters in diameter, and serves to separate the vapor phase from the liquid phase.

4. The fractionating tower (see Figure 86) serves to separate the gases and pressure distillate from the Phlegma (the mixture of gas-oil fractions evaporated in the small furnace together with the fresh gas oil) entering the high-pressure furnace. The tower is 18.8 meters high and 2.74 meters in diameter. It is divided by the baffle plate into two sections, an upper with nine plates and a lower with six. The vapors pass through it as follows: from the evaporator they arrive at the lower section of the tower, pass through the six plates of this section, and when they reach the baffle plate, leave the tower and pass into the exchanger. From the exchanger they are then returned to the upper section of the tower, pass through the 9 plates and leave the tower by the overhead line.

5. The gas separator removes the gas from the pressure distillate. It has three bubble-cap trays, and is 9.14 meters high, with a diameter of 1.52 meters.

6. The exchangers each have a heat-exchange surface of 83 square meters and are used to preheat the mazut by the hot vapors.

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7. The condensers and coolers used in this plant are of the submerged type.

8. The hot-oil pump delivers the phlegma from the bottom of the fractionating column into the high-pressure furnace. The pump has a capacity of 40 cubic meters an hour and operates at a pressure up to 50 atmospheres, pumping the product at 350 degrees C.

9. The reducing valve is designed to reduce the pressure from 45-50 atmospheres in the high-pressure furnace to 3-4 atmospheres at the evaporator.

10. There are charging pumps, used for the reflux, and product pumps.

11. The recirculator recirculates the flue gases in the high-pressure furnace. The cooled gases from the convection chamber are returned by means of the recirculator (a fan of fire-resistant steel) back to the combustion chamber, where they mix with the fresh flue gases and lower their temperature. This favors a gentler heating of the convection tubes and increases their coefficient of heat transfer, since the velocity of the flue gases increases. The operation of the recirculator is a very important factor in the normal cracking process. If it goes out of commission the whole set of operating conditions in a cracking unit is immediately disrupted.

12. Through the recuperator (cold-air fan) cold air is supplied along the channels and is heated to 170 degrees C by the emerging flue gases, which are thereby cooled from 450-500 to 220-250 degrees C. The hot air is supplied to the burners of the furnace.

Flow Chart (Figure 87). The mazut is drawn from the charging

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tank by the pump 1 and is supplied to the low-pressure furnace 3, after first being preheated to 200-250 degrees C in the first exchanger 2 using the heat of the vapors passing from the lower section of the fractionating tower 6 to the upper section, and in the second exchanger 2 using the heat of the vapors passing from the top of the evaporator into the tower. The preheated mazut first enters the convection section of the small furnace, then passes to its radiant section and leaves it after being heated to 400-420 degrees C.

Two streams meet in the mixing T-pipe 15 before the evaporator 5: (1) mazut from the low-pressure furnace and (2) the products of cracking from the high-pressure furnace. The latter leave the high-pressure furnace 4 at 500-505 degrees C. If the cracking products at so high a temperature were allowed to mix with the mazut heated to 400-420 degrees C, then the heavy hydrocarbons contained in the mazut would be cracked, forming a large amount of coke. To stop the cracking reaction the cracking products leaving the high-pressure furnace must be cooled to 420-430 degrees C. For this purpose fresh, cold mazut is pumped into the line after the mixing T-pipe. This process of cooling to stop the cracking reaction is very important for the normal operation of the unit. Thus, the evaporator receives (a) the cracking products from the high-pressure furnace, (b) mazut from the low-pressure furnace and (c) fresh mazut.

The temperature in the evaporator is held within the limits of 415 to 430 degrees C. At this temperature the heaviest tar fractions of the mazut are cracked, forming coke and an additional amount of gasoline. The vapor portion, consisting of (a) gas, (b) pressure distillate, (c) cracked gas-oil fractions and (d)

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straight-run gas oil driven off from the mazut, is separated through the top of the evaporator. The cracked fuel oil is taken off from the bottom of the evaporator, passed through a cooler and run into its tank. The vapors pass through the exchanger 7 and reach the lower part of the fractionating tower at a temperature of 350-365 degrees C. The necessary quantity of fresh gas oil is also pumped into the same place. The vapors pass through the 6 plates of the lower section of the tower and on meeting the baffle plate, pass into the exchanger. They are then returned to the tower and pass through the 9 plates of its upper section, the condenser 7, the final cooler and gas-separator 8, where the cracked gas is separated from the pressure distillate.

From the top of the gas separator the gas is removed into the common line, while the pressure distillate is divided into two streams, one of which enters the reflux of the fractionating tower, and the other the receptacle.

The hot phlegma, cooled to 350-360 degrees C, is collected from the bottom of the fractionating tower by the hot-oil pump 2, which delivers it at a pressure of 45-50 atmospheres to the high-pressure furnace (Figure 85 and 88) for cracking, after it has first passed through the first and second preheaters in the convection chamber, then the tubes of the radiant chamber, and lastly the reaction section (where the cracking of the charge stock takes place). This stock is in the reaction chamber between the first and second preheaters, and the cracking reaction is effectuated in it. The location of the reaction section in the convection chamber makes it possible to maintain smooth and gentle operating conditions without abrupt temperature changes. The furnace-outlet temperature of the cracking products is 500-505 degrees C; these prod-

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ucts then enter the mixing T-pipe. The further course of the products has already been described.

The planned output of a Winkler-Koch plant is 500 tons a day. Our stakhanovites have exceeded this output, and have now in practice reached 650-700 tons a day. The cracking stock consists of 80 percent mazut and 20 percent gas oil pumped into the tower.

At units operating with additional pumping of gas oil, the yield of cracked gasoline reaches 29-30 percent, and, at units without this pumping, 24-25 percent. The yield of gas is 6-8 percent and that of cracked fuel oil is 60-65 percent.

Table 26 gives the approximate operating conditions at a Winkler-Koch unit.

TABLE 26

[1]	[2] Temp., °C	[3] Pressure, atmospheres
Low-pressure furnace:		
(Inlet)	200-250	15 - 17
(Outlet)	400-450	---
Flue gases over the bridge wall	710-715	---
High-pressure furnace:		
(Inlet)	350	45
(Outlet)	500-505	---
Inlet to the reaction section	490	---
Flue gases over the bridge wall	705-710	---
Hot-oil pump	---	35-45
Evaporator	410-420	3.5 - 4

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[1]	[2]	[3]
Fractionating Tower:		
(top)	200-210	3.1 - 3.8
(bottom)	350	.....
Gas separator	---	2.2
Cracked fuel oil (outlet temperature)	120-130	---

The process of recycling the cracking stock and the yield of gasoline at Winkler-Koch units.

The hot phlegma of the fractionating tower consists of a mixture containing the straight-run gas oil distilled at the unit from the mazut, gas oil directly pumped into the tower, and cracked gas oil recycled for cracking from the high-pressure furnace. The latter is termed recycled gas oil, and the whole process is called cracking with recycling. The essence of this process consists in repeatedly returning the gas oil fractions, in which reaction has not gone through to the final stage or in which only part has completed reaction, to the furnace until it has been completely converted into light and heavy hydrocarbons.

As already remarked, when the coke content of the residue is 0.1 percent, the yield of gasoline from the cycle, out of heavy mazut, does not exceed 8-10 percent, while the yield of gasoline from cracking of gas oil amounts to 20-25 percent. The yield of gasoline can be increased if the hield of coke is also increased. However, in this case the unit would be completely clogged by coke after a few days of operation and would be put out of commission.

Another method of increasing the yield of cracked gasoline is employing cracking with recycling, enabling the gasoline yield

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from masut to be increased to 20 percent and that from gas oil to 60 percent, while keeping the coke content in the residue down to 0.1 percent. The more cracked stock is returned for recycle cracking, the higher the yield of gasoline from the fresh feed. But since in this case less fresh feed will enter the unit, the productivity of the unit will consequently fall. In practice, cracking at a recycle ratio of 2 to 3 is considered most advantageous, this ratio meaning the ratio between the sum of fresh and cracked feed and the amount of fresh feed.

Example. A single cracking of gas oil produces 5 percent gas, 20 percent gasoline, 60 percent cracked phlegma and 15 percent cracked fuel-oil. Since the cracked phlegma amounts to 60 percent, 40 percent of fresh feed must be supplied to the unit.

The recycle ratio is $\frac{60+40}{40} = 2.5$.

The yield of products from the fresh gas oil at a recycle ratio of 2.5 is:

Gas	5 x 2.5	= 12.5
Gasoline	20 x 2.5	= 50
Cracked fuel oil	15 x 2.5	= 37.5
	Total	100.0

Faults of the Winkler-Koch unit. Units of this system give a cracked -gasoline yield of 24-29 percent of the feed stock, which is considerably lower than the yield of any other modern cracking plant. This is the principal shortcoming of the Winkler-Koch system. The cracked fuel-oil produced by it is of relatively low viscosity and low specific gravity. Consequently, the light ends remain in the cracked fuel-oil and vanish without trace as cracking stock.

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We note the following faults in the design of this plant:

The small furnace of the plant is not adapted to mild cracking, and its role is confined to heating the feed, thus assuring the separation of the gas oil fractions.

The plant lacks a unit in which the light ends of the cracked-fuel oil could be stripped.

The fractionating tower has an insufficient number of plates and therefore cannot assure sharpness of fractionation.

The heat exchangers are badly designed and frequently go out of service.

73. The Soviet Nefterproyekt System Two-coil Cracking Plant

In 1936 the first Soviet two-coil Nefteproyekt cracking plant was constructed at Grozniy (Figure 89), representing a great forward stride in comparison with the Winkler-Koch plants. The two-coil cracking plant utilized, in part, the flow process and equipment of the latter system, with substantial modifications and additions of which the principal ones were the following:

The small furnace is replaced by one like the high-pressure furnace, which makes possible the mild cracking of mazut;

A second fractionating tower is used, improving the rectification of the cracking products;

An additional evaporator is incorporated to strip the gas-oil fractions from the cracked fuel oil;

A second gas separator is installed; the first one separates the gas from the pressure distillate, the second, from the cracked gasoline;

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A (clay) treating unit is provided for clay treatment of the pressure distillate;

An additional hot-oil pump is provided for delivering the phlegma of the first tower to the mild-cracking furnace;

A stabilizer is provided for stabilizing the gasoline.

The principal equipment of the two-coil Neftеproyekt system cracking plant includes the following aggregates:

1. Heavy-cracking furnace similar in design to the high-pressure furnace. To increase the heating surface of the furnace, a so-called front bank of 12tubes is installed under the burners. The total number of tubes in the furnace is thus brought up to 242. The furnace serves for heavy cracking of the light phlegma from the second tower.

2. The mild cracking furnace is entirely identical in design and dimensions with the heavy-cracking furnace, and serves for the mild cracking of the mazut drawn from the bottom of the first tower.

3. The evaporator differs only in its larger diameter (3 meters).

4. The first fractionating tower serves to separate the heavy ends of the mazut, and the heavy gas-oil fractions already in the furnace, from the vapors of pressure distillate, the light gas-oil fractions entering the furnace and the cracked gases.

The tower has a dual feed: the vapors of the cracking products and mazut feed. It has 9 bubble-cap plates with fluted caps

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for rectification of the vapors, and 5 baffle plates for mixing the feed with the heavy reflux. It is 16.2 meters high and 1 meters in diameter.

5. The second fractionating tower has 12 bubble-cap plates and serves to separate the cracked gas and pressure distillate from the light reflux fed to the heavy-cracking furnace. It is 16.3 meters high and 3 meters in diameter.

6. The gas separators. The first gas separator separates the gas from the pressure distillate, and the second one from the finished cracked gasoline.

7. The additional evaporator (Figure 91) serves to evaporate the heavy solar fractions from the cracked fuel oil, and simultaneously for preheating the mazut. It consists of an upper section with four bubble-cap plates and a lower (evaporation) section with four baffle plates.

8. The absorber is designed to free the gas from the light ends of the pressure distillate entrained with it. It has twenty plates with round caps.

9. Four feed heat exchangers preheat the mazut charge by using the heat of the reflux from the second column. The heating surface of each exchanger is 74 square meters.

10. The condenser consists of two parts and condenses the pressure distillate and the finished cracked gasoline.

11. By means of the cooler, the cracked fuel oil and reflux cool the cracked fuel and cracked reflux from the second tower. For this purpose it is divided into two coils mounted in a single tank.

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12. There are two hot-oil pumps, one from the Sumsk plant delivering reflux from the second tower to the high-cracking furnace, and the other an American Transit pump delivering reflux from the first tower to the primary cracking furnace. Experience has shown the former to operate better than the latter.

The flow sheet of the Nefteproyekt system two-coil cracking plant is given in Figure 92.

For better feed utilization in these plants, in contrast to Winkler-Koch plants, the mazut is delivered not to the furnace but to the upper section of the additional evaporator 11. The charge first passes through the four exchangers 15, where it is preheated to 80-100 degrees C by using the heat of the reflux from the second tower. The cracking residue is passed from evaporator 3 into the lower section of the additional evaporator on account of the reduction in pressure from 5-6 atmospheres in evaporator 3 to 1 atmosphere in the additional evaporator. The solar fractions are evaporated out of the cracking residue because of the sharp fall in pressure, pass to the upper section of the additional evaporator and condense into the mazut there, raising its temperature to 100-110 degrees C. The preheated mazut, thus enriched by the solar fractions, now passes into the first fractionating tower 4 on to three plates: the bottom knockout plate and the fifth and seventh bubble-cap plates. The vapors of the cracked gasoline, light solar, heavy solar and cracking gas pass into the same tower underneath the bottom plate. The heavy solar condenses and mixes with the mazut, while all the other products, together with the light fractions stripped from the mazut (at 360-370 degrees C) pass to the second tower, 5. The reflux from the first column 4, consisting of

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heavy solar and mazut at 350-360 degrees C, are gathered by the Transit hot-oil pump and delivered at a pressure of 40 atmospheres to the primary cracking furnace for light cracking.

The primary cracking products leave the furnace heated to 465-475 degrees C, pass a reducing valve and mix with cold mazut, and then, already somewhat cooled (to 420-430 degrees C) they enter the evaporator.

The vapors of the pressure distillate and light gas oil, together with the cracking gas, enter the second fractionating column. The vapors leaving this column at 240-250 degrees C are divided into two streams: one passes through the pressure-distillate condenser 6 to the gas separator 8, whence it is returned, completely free of cracking gas, as reflux to the second tower; the other stream is directed to the clay treatment unit. The course of this second stream is traced below.

The reflux of the second fractionating tower 5 consists of: (1) the straight-run, gas-oil fractions distilled off from the mazut in the first column, (2) the cracked gas-oil fractions obtained from the primary cracking of mazut, (3) the heavy-cracked feed from the heavy-cracking furnace.

Part of the phlegma enters the feed heat-exchangers 15 to preheat the mazut charge, passes to the coolers and then to the reflux of the first column. The remainder of the phlegma enters the admission line of the Sumsk hot-oil pump at 320-330 degrees C and is delivered to the high-cracking furnace 2 under a pressure of 50 atmospheres. The cracking products leave this furnace at 500-502 degrees C, are cooled by a steeam of cold product to 420-430 degrees C, and then enter the evaporator. Here the vapors are

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separated from the liquid and pass to the first tower, while the liquid phase, as already stated, passes to the additional evaporator. The latter allows the use of watery mazut as feed, since the water is evaporated on the upper plate and then goes out of the system into the condenser 12, whence it flows to a tank. Steam is fed into the bottom of the additional evaporator to strip the cracking residue of the light gas-oil fractions. This residue, now brought to a viscosity of $E_{50} = 80$, is pumped from the bottom of the additional evaporator through a cooler into storage.

Let us now trace the path of the pressure distillate in the vapor-phase treatment (Figure 93).

The pressure distillate contains a large amount of unsaturated hydrocarbons. Part of them are very unstable, are readily polymerized; and converted into heavy polymers, and partially resinify. For this reason raw cracked gasoline in storage rapidly forms gum, darkens and becomes unsuitable for use.

The treatment towers 2, charged with a special clay (up to 25 tons per tower), are employed in Nefteproyekt-system cracking plants. This clay, of the Gumbri deposit (Georgian SSR), which is termed Gumbri clay, has the property of retaining (adsorbing) resins on its surfaces, while the unstable hydrocarbons are converted to polymers. Most of the cracked gasoline passes through the clay without change. The vapors of the pressure distillate from the second tower 1 pass through one of the treatment towers 2 (the second of these towers being under repair or in reserve) and then to the additional tower 3 for fractionating the purified pressure distillate (see Section 117).

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The temperature of 225-240 degrees C at the top of the column assures production of a merchantable cracked gasoline conforming to the standard. The polymer products formed in the treatment towers also enter the same column for removal of their light ends. The polymers, which are used as fuel or as feed for the secondary cracking, flow from the bottom of the column through the cooler 7. The cracked-gasoline vapors and the cracked gas pass from the top of the column into the condenser 4, the second gas separator 6, and then to the stabilizing unit.

The gas is separated from the cracked residue in the gas separator, in which no rectification takes place. Consequently, the gas carries over with it a considerable quantity of light-gasoline fractions, while part of the gas remains in a state of solution in the cracked gasoline (propane, propylene, butane, butylene).

On storage of this cracked gasoline, the gases dissolved in it are evaporated and carry light gasoline fractions with them, thus causing a loss of the gasoline and deterioration in its quality.

For this reason the two-coil cracking plants employ a gasoline stabilization process which consists of its fractionation thus separating the dissolved gases.

The stabilization plant represents a continuation of the cracking plant and the cracked gasoline is routed to it after its vapor-phase treatment.

The stabilization plant consists of:

(a) a stabilizer, i.e., a rectifying bubble-cap tower with 20 plates, designed to operate under 40 atmospheres pressure;

(b) exchangers and

(c) a stripping still, which is a unit for stripping the light ends from the gasoline.

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Figure 94 shows the flow diagram of the stabilization plant. The cracked gasoline from the washing tower is collected in the receptacle 1, whence it is pumped through the exchangers 8 into the stabilizer 1.

The exchangers are heated by the cracked gasoline leaving the stabilizer. The stabilizer works on the heat of the circulating phlegma of the second tower of the cracking plant, which is delivered to the stripping still. The liquid passes from the lower plates of the stabilizer to the stripping still 6, is heated, and then returned under the first plate. The preheated cracked gasoline evaporates its light ends, which ascend through the stabilizer. The column is refluxed by a mixture of propane, propylene, butane and butylene. From the top of the column the product passes through the condenser-cooler 7 into the reflux tank. The uncondensed part of the product passes to the burners for burning. The stabilized gasoline passes through the exchangers 8 and the coolers 5 into storage.

To separate the light gasoline fractions from the gas, the gas is routed from the gas separator to the absorbing column (absorber).

A Nefteproyekt plant cracks about 850-900 tons of mazut a day, yielding:

gasoline 34-37 percent

cracked fuel oil 55-58 percent

gas 7-8 percent.

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TABLE 27

APPROXIMATE PROCESS CHART OF THE TWO-COIL CRACKING PLANT

Unit	Control points	Temperature, degrees C from	to	Pressure, atmospheres from	to	Number of strokes from	to	Level (test cocks) from	to
[1]	[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]	[10]
Mild-cracking furnace	Outlet of radiant section	452	454	-	-	-	-	-	-
	Outlet from reaction section	474	475	-	-	-	-	-	-
	Flue gases over bridge wall	---	690	-	-	-	-	-	-
	Hot-oil pump	---	---	47	50	48	49	-	-
Evaporator	Top	-	420	5	5.2	-	-	2	3
	Bottom	427	430	-	-	-	-	-	-
Additional evaporator	Top	-	-	-	-	-	-	2-3	1-2
	Middle	210	220	-	-	-	-	-	-
	Bottom	360	365	0.5	0.8	-	-	1-2	2-3

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TABLE 27

[1]	[2]	[3]	[4]	[5]	[6]	[7]	[8]	[9]	[10]
Cracking-plant cooler	Outlet	105	108	-	-	-	-	-	-
First Column	Top	363	365	-	-	-	-	-	-
	Bottom	375	380	4.7	5	-	-	3-4	4-5
Second column	Top	245	250	-	-	-	-	-	-
	Bottom	330	335	4.3	4.7	-	-	3-4	4-5
Treatment tower	No 1	-	225	-	-	-	-	-	-
	No 2	-	225	3.9	4.1	-	-	-	-
Additional column	Top	185	190						
	Bottom	205	210	3.7	3.7	-	-	1-2	2-3
Gas Separators	No 1	-	-	2.7	3	-	-	-	½
	No 2	-	-	2.7	3	-	-	-	½

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74. The Combination Four-coil Cracking Plant

Plants that perform the integral processing of crude oil from distillation to cracking the distillates and residues producing (1) straight-run, cracked and reformed gasolines, (2) gas and (3) cracked fuel oil, are termed combination plants.

Combination plants combine the processes of primary distillation of crude, mild cracking of mazut, selective cracking of cracking stock, reforming of ligroin and stabilization of the cracked gasoline. To effectuate all these processes combination plants have 3 to 5 furnaces, a certain number of columns and evaporators and a large number of exchangers. Such a plant is highly efficient since the waste heat is excellently utilized; it occupies relatively little space and gives high yields of cracked gasoline.

Let us familiarize ourselves with one of these plants, constructed in the USSR, the four-coil combined cracking plant of Figure 95, with a capacity of 3,000 tons a day.

Selective cracking is performed in this plant. What is selective cracking? If a mixture of heavy and light fractions, of fresh feed and recycled feed, is cracked in a furnace under some intermediate set of operating conditions, then this set will be too high for the heavy hydrocarbons and too low for the light ones. The advantage of selective cracking consists exactly in the division of the feed into several close-cut fractions, each of which is cracked in a separate furnace under the optimum operating conditions specially selected for it. In the plant to be described, the residue from straight-run distillation and the heavy, already cracked fractions are subjected to mild cracking in the first fur-

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nace (mild cracking furnace); in the second furnace, straight-run gas oil and the cracked fractions from the mild cracking furnace are cracked; in the third, the cracked fractions from the second furnace are cracked; in the fourth, reforming of ligroin takes place.

This method of cracking gives high gasoline yields (for instance, 51 percent of gasoline and 1 percent of gas is produced from Ishimbay crude in this plant).

Processes carried out in the plant are: (1) straight-run distillation of crude, giving gasoline, ligroin and residue; (2) reforming of ligroin; (3) mild cracking of mazut mixed with heavy cracked fractions, in the mild cracking furnace; (4) cracking straight-run gas oil, mixed with mild-cracking gas oil; (5) cracking the cracked fractions from the gas-oil furnace, in the reforming furnace, together with the already cracked fractions of this furnace; (6) stabilization of the straight-run gasoline together with the cracked and reformed distillates.

As will be realized from this recital of the various processes, the four-coil plant is very complicated, and withal, all of its separate parts must operate continuously and mutually coordinated.

All of the furnaces in this plant, including that for mild cracking (Figure 96), are distinguished by their dimensions (length 10.6-11.9 meters, width 11.8-16.7 meters, height 6.1-6.9 meters) and by their structure. The mild-cracking furnace has three chambers: radiant, reaction and convection. The radiant and reaction sections are separated by a bridge wall; the convection chamber is placed on top and is separated from the radiant and reaction chambers by a flame arch. The side-tube banks of the radiant and reaction

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chambers have a single row of tubes, while the roof-tube banks have two rows. All the other furnaces are built in the same way.

The straight-run residue, together with the recycled fa fractions from the mild-cracking furnace, are cracked in the same furnace. This mixture is gathered from the tanks of column D-2 (see Figure 95) at 371 degrees C by the hot-oil pump C-7 and delivered to the furnace under 28 atmospheres pressure.

The gas-oil cracking furnace is designed to crack the straight-run and the mild-cracked gas oils, which are gathered at 232 degrees C from the tank F-5 by the hot-oil pump G-18 and delivered to the furnace under 43 atmospheres pressure.

The recycle-cracking furnace performs two processes: (a) preheating the crude oil (after the exchangers) from 260 to 288 degrees C in the convection chamber, and (b) cracking the phlegma from column D-4, representing the cracked and recycled fractions from the second furnace.

The reforming furnace is designed to reform the ligroin delivered by the hot-oil pump G-3 from the tank of column D-1 at 93 degrees C, under 70 atmospheres pressure.

The rectification column D-1 separates the crude into light naphtha, ligroin and semi-mazut. It consists of two sections: the lower or evaporator section with 7 bubble-cap trays separates the light fractions from the semi-mazut; the upper or column section has 10 bubble-cap trays and separates the light fractions arriving from the evaporator.

The rectification column D-2 also consists of two sections:

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the lower or evaporator section has 3 bubble-cap trays and separates the light fractions from the cracking residue and the reformed ligroin; the middle section has 9 bubble-cap trays and separates the semi-mazut from the gas oil and mazut; the upper or rectifying section has 6 trays and rectifies the mild-cracked gasoline.

The rectification column D-4 also consists of two sections: the lower or evaporator section is designed to separate the cracking residue from the products of cracking and reforming, while in the upper or rectifying section the final rectification of the pressure distillate is performed and the recycle fractions are collected for the recycle cracking furnace.

The cracking residues leaving the evaporator of columns D-2 and D4 still contain a considerable quantity of gas-oil fractions, which are stripped in the stripping column D-3 by passing steam into the residues and reducing the pressure to 0.15 atmosphere.

The light C_2, C_3 and C_4 hydrocarbons are diven off from the pressure distillate in the stabilizer-absorber D-5. In the absorber the light gasoline fractions are absorbed from the gases leaving the gas unit.

The stabilizer D-6 stabilizes the straight-run gasoline. The gravity settler F-9 separates the water and sediment from the preheated crude.

The gas separator F-2 separates the gases from the mild-cracked naphtha fractions.

In addition to these units, the plant is also equipped with

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accumulators (storage tanks), condensers, coolers, pumps, control instruments, etc. It has a large number of automatic controllers of temperature, pressure, liquid level, and of meters, etc.

The Flow Sheet of the Selective Cracking Plant (See Figure 95)

Ishimbay crude, which usually contains salts, sediment and water, is used as the feed stock for this plant. For this reason it is first desalted and dehydrated at the producing lot. After the crude has been preheated to 115 degrees C in the gas-oil heat exchanger E-3 at the cracking plant, it is subjected to a supplementary gravity settling in the dehydrator F-9. The crude receives the necessary heat for its evaporation from the exchangers E-10 and E-13 (in which it is heated to 265 degrees C by the heat of the circulating reflux) and in the tubes of the convection section of the recycle cracking furnace, where it is heated to 288 degrees C. The heated crude is then evaporated in the straight-run still D-1 and is separated into (1) light gasoline and gas, (2) ligroin and (3) semi-mazut.

The products obtained from the still D-1 then take the following route. The light gasoline is condensed, separated from the gas in the gas separator F-6, and freed from the light ends in the stabilizer D-6. After cooling, the stable gasoline is routed to storage, while its unstable fraction is used as reflux for the stabilizer.

The ligroin is the feed for the reforming furnace, to which it is delivered under 53 atmospheres pressure by the pump G-3 from the tank of the still D-1. The reforming is effectuated at 510 degrees C. The products of reforming, cooled by the cracking phlegma, enter the evaporator of column D-4.

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The semi-mazut constitutes a very wide fraction and cannot be used as cracking stock. It is therefore delivered to the 15th plate of the low-pressure column D-2 (pressure 5 atmospheres) for fractionation into closer-cut fractions.

The System of Mild Cracking and the Cracking of Gas Oil.

Besides semi-mazut, the column D-2 also receives products from the mild-cracking furance and the cracking residues from the column D-4. It receives (a) the mild-cracking naphtha fraction and gas, (b) a broad fraction for cracking, (c) a fraction for the mild-cracking furnace and (d) the residue after mild cracking and cracking.

The fractions received in column D-2 are then routed as follows:

The light naphtha fractions and gas go from the top of the column to the gas separator F2 for separation; the gas passes to the gas fractioning unit and the distillate to column D-4.

The gas-oil fraction, which is feed for the cracking furnace, flows from the 10th plate of the column into the tank F-5. Part of the gas oil is used to cool the products of cracking, while another part is collected by the pump G-18 and delivered under 18 atmospheres pressure to the furnace for cracking. The products of cracking leave the furnace at 610 degrees C, are cooled by the cracking phlegma, and routed to the evaporator of the high-pressure column D-4.

The pump G-7 collects the feed for the mild cracking from the middle bottom of the column D-2 at 360 degrees C and delivers it to the furance under 17.5 atmospheres pressure; the products of

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mild cracking leave the furnace at 468 degrees C and enter the evaporator of column D-2.

The cracking residue is bypassed from the lower section of the column into the additional evaporator D-3 (operating under a pressure of 0.15 atmosphere) for stripping the solar fractions, which are returned to column D-2 as feed for the mild cracking furnace, while the cracking residue is pumped into storage through the cooler.

The System of Recycle Cracking

The phlegma from column D-4 serves as the feed for the recycle cracking furnace. In this high-pressure column D-4, the cracking products from three furnaces are gathered: the gas-oil cracking furnace, the reforming furnace, and the recycle cracking furnace. These products are separated into three portions: (1) pressure distillate, (2) cracking phlegma, and (3) cracking residue.

(1) The pressure distillate and gas are led off from the top of the column into the stabilizer-absorber D-5 for separating the gas and stabilizing the pressure distillate.

(2) The cracking phlegma is collected at 371 degrees C from the middle bottom of the column by the pump G-15 and delivered to the recycle cracking furnace under 53 atmospheres pressure. The products of cracking leave the furnace at 524 degrees C, are cooled by the cracking phlegma, and enter the evaporator of the straight-run still D-1.

(3) The cracking residue passes to column D-2.

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The reforming system at the present time has been separated and set up as an independent process, and ligroin may be reformed independently of the operation of the rest of the plant. In this case ligroin from the straight-run still D-1 is collected in the ligroin storage tank, from which it is pumped through an exchanger into the reforming furnace. Four additional exchangers are provided for preheating the ligroin. The products of reforming, mixed with the cooling product, ligroin, enter the evaporation section of column D-7 (not shown on the flow sheet). The residue from column D-7 is routed to the evaporator of column D-2. The gasoline vapors and the gas pass from column D-7 to the gas-separator F-10 for separation.

Thus, the selective cracking plant may operate either on the complete cycle (including the reforming furnace) or on an incomplete cycle (without the reforming furnace). The latter is done in the event of the reforming system needing repair, or if insufficient ligroin is yielded owing to some operating trouble in the plant.

The following products (in %) are obtained from Ishimbay crude by the Lummus plant, with its subsidiary units:

Light straight-run gasoline	5-7.5	
Reformed gasoline	14 - 16	Total gasoline 43-51.5
Cracked gasoline	24 - 28	
Cracking residue (boiler fuel)	29 - 30	
Gas	14.5	
Hydrogen sulfide	0.5	
(Losses 2.5).		

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75. The Two-Coil Selective Cracking Process with Reaction Chamber

The two-coil selective cracking system (Figure 97) differs from the other systems we have considered by the presence of an independent reaction chamber, as well as in the design of the furnace.

The tubes in the furnace of this plant (Figure 98) are arranged in two vertical rows in narrow chambers. The burners are placed on the top, and the flames directed downwards. Each tube of the furnace is heated uniformly by radiation from both sides. The heat liberation of the tube surfaces reaches 55,000 to 60,000 calories per square meter per hour instead of the 30,000 to 35,000 calories which is the maximum attainable with furnaces of ordinary design.

The furnace of this plant has no internal reaction section, and the reaction takes place in a special reaction chamber instead, which consists of a hollow steel cylinder of height up to 12 meters, 3 meters in diameter, designed for high-pressure operation. Coke is deposited in this chamber and is periodically cleaned out (during the repair periods of the plant).

Figure 99 shows the flow sheet of the plant. The feed pump delivers the charge around the middle of the column 5. The vapors of the cracking products enter the column at a point somewhat below this. The charge flowing downwards meets these vapors and is heated by them to 420 degrees Centigrade, while the cracking vapors themselves are accordingly cooled. The volatile fraction of the charge is vaporized thereby, and ascends in the column, while the heavy fraction of the cracking vapors is condensed,

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mixes with the residue of the charge, at the bottom of the column, and is collected in the form of a heavy charge for recycle cracking. The hot-oil pump delivers this heavy charge for recycle cracking through the heater 1, where it is subjected to the action of high temperature and pressure. A mixture of the kerosene and gas oil fractions of the charge and cracking products is drawn out of the column from a point above the charge inlet. This mixture is delivered by a hot-oil pump through a second heater 2, where it is heated to higher temperatures and subjected to higher pressures than those in the first heater. On emerging from the heaters, the two streams of cracking products merge into one and enter the reaction chamber 3, where they are subjected to a temperature of up to 495 degrees Centigrade and a pressure of 10-20 atmospheres. Here the cracking takes place without additional heat being supplied from an outside source.

The cracking process yields cracked gases, cracked gasoline, cracked gas oil, cracked fuel oil and coke.

The coke accumulates in the reaction chamber, while all the separate products pass in vapor form into the additional evaporator 4, operating under reduced pressure.

The cracked fuel oil is drained out of the bottom of the additional evaporator, while the vapors emerging upwards pass into a fractionating column, first passing through a heat exchanger where they preheat the feed for the cracking furnace.

In the column, as usual, heavy and light products for mild cracking are separated from the vapors of the reaction products. The vapors of the cracked gasoline, and the gas, pass from the top

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of the column into a condenser and then into a stabiliser, where the gas is taken off, and finally the stabilised cracked gasoline is routed to storage.

The plant can be operated according to two schemes: all the way to coke formation, or with the process continued only to a liquid residue.

In the former case a large amount of coke is formed (up to 28 percent), which clogs up the chamber in 40-100 hours on stream, thus forcing the plant to be taken off stream to clean out the coke. In this case the gasoline yield is higher than when operating only to a liquid residue.

In the latter case the yield of coke does not exceed 0.1 percent, and the plant can operate for 18 to 36 days, depending on the quality of the feed. Comparative data on the operation of two-coil systems on both schemes are given below.

Table 28

MATERIAL BALANCE OF CRACKING PLANT

	Operating:	
	To Coke Formation	To Liquid Residue
Yield of motor gasoline (in %)		
Mazut feed	Up to 40	Up to 30
Gas-oil feed	Up to 65	Up to 50-60
Yield of coke in %	10 - 28	0.03 - 0.10
Yield of cracked fuel-oil in %	10 - 19	14 - 59
Yield of cracked gas, in %	6 - 17	8 - 10
Temperature, degrees C	495 - 505	490 - 520
Pressure in chamber, atmospheres	--	11 - 20
Stream time of plant	40 - 100 hours	19 - 32 days

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76. Cracking with the Addition of Gases

Cracking of a liquid charge stock with the addition of gases represents an advance over thermal cracking. This process is conducted in the presence of the following gaseous hydrocarbons: propane, propylene, butane and butylene. It makes it possible both to increase the yield of gasoline (by a few percent) and its octane number (by 3-6 points). The process takes place at higher temperatures and pressures than the cracking systems we have just described, and without increased coke formation.

One of the favorable features of this process is the possibility of producing an aviation fuel of 80 octane number.

The equipment used for this process differs in no way from that for thermal cracking, but is heavier, since the process involves higher pressures.

Various feeds may be used, ranging from ligroin to mazut. Figure 100 is a schematic flow diagram of the process, operating on a ligroin feed.

Ligroin enters the top of the absorber 7 and flows downward to meet the ascending cracking gas. The light gaseous hydrocarbons (C_1, C_2) are eliminated from the absorber, while the heavier ones (C_3, C_4) dissolve in the ligroin. The ligroin, enriched by the gaseous hydrocarbons, is pumped through the furnace 1. On leaving the furnace, the products of cracking encounter the residue, delivered to produce abrupt cooling and terminate the reaction. This product consists of the residue leaving the column and circulating through the cooler. The cooled reaction products enter the fractionating tower 2.

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The operation of the tower is controlled by the circulating reflux 3 at the top, and by the circulating gas-oil reflux 4. The bottoms from the tower are removed, and part of them used for cooling. The vapors from the top of the tower are passed through the stabilizer 6. The bottoms, from the stabilizer, consist of stabilized gasoline, while the cracking gas is removed overhead and passed through an absorber, adding gas from an external source into the gas line in the quantity required for the process.

Operating conditions: feed. Ligroin, gas oil, topped crude or mazut may be used as feed for this process. The gas arriving at the plant consists mainly of the propane-propylene and butane-butylene fractions. It is added in the amount of 25 percent to 150 percent of the feed, with the minimum amount going to a mazut feed and the maximum to a ligroin.

Temperature and pressure. The presence of large quantities of gaseous hydrocarbons requires increased pressures in both furnace and tower, and at the same time permits a higher temperature to be maintained in the furnace than those allowable in thermal cracking furnaces. This temperature depends on the composition of the feed and the characteristics of the finished product, and ordinarily ranges from 550 to 605 degrees Centigrade. The pressure varies between 70 and 136 atmospheres. Such high pressures are maintained to keep the feed in the liquid phase at all times and to keep it physically uniform, to prevent increased coking.

Yields and characteristics of the products. In operation on a ligroin feed, the following operating conditions are maintained:

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Furnace-outlet temperature	530-590 degrees C
Pressure at furnace outlet	70-105 atmospheres
Pressure in the tower	21-28 atmospheres

Under these conditions about 40 percent of ordinary aviation fuel, along with 35 percent of so-called safety aviation fuel of the following characteristics is produced:

TABLE 29

	Ordinary Aviation Fuel	Safety Aviation Fuel
Specific gravity	0.756	0.845
Octane number, unblended	77	80
Octane number with 3 cc of ethyl fluid	90	88
Initial boiling point, degrees C	49	149
50% evaporated to degrees C	96	165
End Point, degrees C	152	220

When a heavy feed is cracked in the presence of gases, the yield of motor gasoline is considerably increased over that of thermal cracking (using topped crude of specific gravity 0.930 as feed):

TABLE 30

	Thermal Cracking	Gas Reversion Cracking
Temperature, degrees C	482	530
Pressure, atmospheres	14	53
Octane number of treated gasoline	66	70

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77. The Normal Operation of Cracking Plants and the Control and Adjustment of Their Operating Conditions

Cracking plants operate under very rigid operating conditions, and thus even insignificant deviations from the prescribed conditions may lead to reduction in the gasoline yield or to a slight coking, which will be reflected in the duration of the working cycle. More considerable departure from the process chart, for instance, an increase (jump) in the high-pressure furnace-outlet temperature of 10-15 degrees, will result in rapid coking of the furnace tubes and shutdown of the plant. For this reason the prescribed operating conditions of a cracking plant must be smoothly and gently maintained, and no deviations allowed. In doing this the attention of the attendants must be fixed primarily on the readings of the control instruments. Production troubles and the methods of overcoming them, in connection with breaches of the prescribed operating conditions, will be discussed in Section 79, but here we limit ourselves to a description of the normal operation of Winkler-Koch and cracking plants.

1. The furnace serves not only as an apparatus for heating the feed, but also as the reaction chamber in which the process of cracking takes place. Cracking is commenced in the radiant section of the furnace and is completed in the reaction section, with the temperature in the latter rising only 8 - 10 degrees C, since almost all of the heat received is used up in it on the cracking reaction. To maintain a gentle and steady set of operating conditions in the reaction section, as has already been stated, this section is located in the convection chamber between the first and second preheaters.

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The basic indices that must be watched all the time the furnace is operating are the feed temperatures on leaving the radiant and reaction sections, the difference between the two, the temperature of the flue gases over the bridge wall and the furnace inlet and outlet pressures.

The furnace-outlet temperature is controlled: (1) by the flue gas temperature over the bridge wall, (2) by the rate of feed of the charge (by changing the number of strokes of the hot-oil pump) and (3) by the pressure inside the furnace.

Assume that the feed temperature at the outlet from the reaction section must be raised 2-3 degrees C. The flue-gas temperature is controlled automatically and is held constant at its upper limit, for which reason the furnace-outlet temperature is controlled by the number of strokes of the pump or by changing the pressure at the outlet of the furnace.

The furnace-outlet temperature may be reduced by the opposite operation. It is very important to hold the difference between the outlet temperature of the radiant section and that of the reaction section constant, at between 8 and 10 degrees C, throughout the entire cycle. Any fall in this difference indicates abnormal operation of the hot-gas fan or incipient coking of the furnace tubes. In the former case the fan operation must be adjusted, and in the latter the outlet-temperature of the radiant section must be somewhat reduced.

The pressure in the furnace depends on the furnace-outlet temperature and on the number of strokes of the hot-oil pump.

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If the furnace-outlet temperature and the number of strokes of the hot-oil pump are both normal, then an increase in pressure may be ascribed to coking of the reducing valve. If the pressure still does not drop when this valve is opened, it follows that the furnace coil or the transfer line from the furnace to the evaporator is obstructed by coke.

2. During the operation of the low-pressure furnace, the outlet-temperature of the feed must be watched. This value depends on the flue-gas temperature over the bridge wall and on the quantity of the charge being fed into the furnace. The flue-gas temperature is controlled by the feed of fuel into the burners, and this rate of fuel feed is in turn controlled by changing the number of strokes of the charging pump.

3. The normal operation of the evaporator is mainly determined by the specific gravity of the cracked residue, which in turn depends on the temperature in the evaporator and on the level of the product in it. The temperature in the evaporator is usually followed by a thermocouple installed on top of it, since one installed inside it is rapidly coked up and does not give correct readings. The level in the evaporator is determined by the seven testcocks.

The following operating conditions are established for the evaporator. The temperature at the top is held at 420-425 degrees C, and the level of the product kept between the 2nd and 3rd test cocks. Increasing the level of the product in the evaporator leads to prolonging the time required to process it, as a result of which more of the gas-oil fractions are evaporated off and the cracking

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reaction thus proceeds more energetically. An increase in the specific gravity of the cracking residue will result. An increase in the evaporator temperature will also cause more of the gas-oil fractions to be evaporated, accelerate the cracking reaction and increase the specific gravity of the cracking residue. A temperature rise in the evaporator may be due to a rise in the furnace-outlet temperature or a reduction in the rate of mazut pumping into the mixing tee. The evaporator temperature may be easily regulated by pumping in cold product. In this way, to assure normal operation of the evaporator, the specific gravity of the cracking residue must be watched, together with the temperature at the top of the evaporator and the level of product in it.

4. The operation of the rectification column is judged by the temperature of its top and bottom and on the level of the product in it. The temperature of the top of the second column is automatically controlled by pumping in pressure-distillate for reflux. The temperature is established according to the characteristics of the pressure distillate to be produced. The temperature of the bottom of the column is controlled by pumping in cold gas oil. The temperature of the top of the first column is regulated by the supply of reflux, while the temperature of the bottom is controlled by the plate at which the feed is pumped in.

The level in the columns is controlled by test cocks, as it is in the evaporator. The normal level in a column must be maintained because the lower section of the columns serve as the reservoir for the feed to be taken by the hot-oil pump for delivery to the furnace. Any deviation from this normal level will be immediately reflected in the operation of the furnace. The level in

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the first column is maintained by pumping in the necessary quantity of feed stock, but that in the second column (and likewise in the column of the Winkler-Koch plant) is maintained by pumping in gas oil. If it becomes necessary to lower the level in the column, part of the phlegma will be drawn off, while if it must be raised, the draining of the phlegma is stopped. The level in the first column (and in the Winkler-Koch column as well) also depends on the evaporator temperature: when this rises, the vaporization of the solar fractions is intensified and the level in the column is therefore increased; while a lowering of the level in the column is reflected in the delivery of product to the furnace. Increase of the level above the normal is also harmful, since it will not in that case be possible to evaporate the tailings of the pressure-distillate out of the phlegma, and this will lead to reduction in the yield of gasoline and in the useful charge of the furnace, since the gasoline and kerosene fractions are not cracked under the operating conditions of a cracking plant and thus constitute ballast. For this reason the level in the column must be held constant between the 2nd and 3rd test cocks.

The color of the phlegma arriving in the high-cracking furnace (and also in the high-pressure furnace) must be checked as often as possible. If the phlegma has darkened this means that feed stock has gotten into it (leakage of an exchanger). In this case immediate steps must be taken to eliminate the causes of darkening in the phlegma.

5. In the operation of the gas separator, the level of the liquid is watched (by the aid of the test cocks), as well as the pressure, which is automatically controlled by the valve or gate valve on the gas line.

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6. The viscosity of the cracking residue sharply rises as the temperature falls; and at normal temperatures it can be pumped by a pump only at fairly high pressures. In the Winkler-Koch plant, the cracking residue leaves the evaporator by gravity flow (under 5-6 atmospheres pressure). If the discharge of this residue is not to be impeded, its viscosity must not go beyond the allowable limits. For this purpose the water in the cracking-residue cooler is kept at boiling temperature. If the temperature of the cooling water should drop, the cooler might, as they say, tar up, and the discharge of cracking residue from it stop. Coke gradually accumulates in the tubes of the cracking-residue cooler, and the discharge of the residue is thus hindered. The cooler is therefore systematically flushed with solar oil pumped through it, but the pressure on the pump must not go over 30 atmospheres.

The operation of the pumps is controlled by the number of their strokes, which is prescribed by the process chart. There is an emergency pump for every pump at a plant (except for the hot-oil pump). For this reason, as soon as a defect in a pump is noticed it is turned off and the emergency pump turned on to replace it. There must be an especially careful and continuous surveillance over the hot-oil pump.

The laboratory control of cracking plants. At cracking plants a control is maintained over the properties of the feed, the phlegma, the cracking residue, the pressure distillate and the cracked gasoline (every hour). The properties of the cracking residue are kept under especially careful observation: its specific gravity is determined, and, if any deviations from the norm are detected, the results of the analysis are immediately telephoned to the plant.

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The content of gas oil fractions in the cracking residue is periodically determined (by Engler distillation) as well as the content of carboids, the maximum value of which must not exceed 0.1 percent. Increased gas-oil content in the residue indicates slack operating conditions in the evaporator; in this case the temperature or the level of the liquid in it must immediately be raised. Increase in the carboid content indicates too tense operating conditions; and either the temperature of the evaporator or the level of the liquid in it must be lowered.

The cracking phlegma is taken from the lowest test cock of the rectification column. The color of the phlegma is determined visually at the plant, while its specific gravity and fractional composition are determined at the laboratory. Increased content of the light fractions in the phlegma shows that it contains kerosene and gasoline fractions. The harmfulness of these fractions in the cracking residue has already been mentioned -- they are useless ballast. After distillate is drawn off from the pumps or the gas separator, its specific weight and fractional composition is determined. If this product is too heavy, the delivery of reflux to the top of the column is increased, while if it is too light, the reflux is diminished.

78. Production Troubles and Possible Accidents in the Operation of a Cracking Plant

Violation of the process operating conditions and production troubles are more serious in the operation of a cracking plant than in that of a plant for the distillation of crude oil and mazut, since cracking plants operate under very tense conditions. Violations of the prescribed technical rules lead to accidents often accompanied by fires.

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The safety rules at cracking plants are of especially great importance since these plants operate under high pressures and at high temperatures. As a result of the natural wear of the materials of which equipment, piping and unions are made, hot liquids may be thrown out of the apparatus (for instance, when a tube burns out) causing fire and accidents.

Most accidents are the result of infraction of the rules for servicing a plant. Special attention must therefore be paid to the operation of the apparatus at a cracking plant, and the rules of technical safety and fire protection carefully observed.

Let us consider the most characteristic production troubles and possible accidents that can occur during the operation of a cracking plant.

1. Coking of the furnace tubes. Coke is formed to a varying extent in the different sections of a furnace. It is deposited in small amounts in the preheaters. In the reaction section a cracking reaction takes place, and therefore considerably more coke is deposited in it. The temperature is lower in the radiant section than in the reaction section, but in spite of this much coke is formed, since the tubes of this section operate under very severe conditions. If a plant is operating normally, coke is deposited gradually on the walls of the tubes. If there is a sharp jump in temperature (whatever the reason may be) coke is formed much more rapidly and may soon block up all the tubes.

Jumps in temperature may occur in case:

(a) gasoline from the gas separator enters the burners together

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with the gas; (b) the hot-oil pump stops, or slows down, resulting in stopping the feed of the charge into the furnace, while the stay of the charge in the furnace is consequently prolonged. The hot-oil pump may break down and stop, or it may be slowed down as a result of an undue rise in the level in the rectifying column. Coking of the furnace may also occur if any foreign object remains in the tubes (a bolt, a chipping hammer, etc).

The formation of coke in a tube may be recognized by the appearance of dark spots on the tubes, and by the increase in the pressure differential between the inlet and outlet of the furnace. The pressure-differential may rise from 14-15 to 20-22 atmospheres if there is coking. The reducing valve is usually completely shut at such a time and the pressure can therefore not be controlled by manipulating it.

Increased coking of the furnace tubes thus leads to the plant going prematurely off stream.

2. Burning of the furnace tubes. The tubes in a cracking furnace operate under very severe conditions -- at high temperatures and pressures, and in an oxidizing medium, since the flue gases contain free oxygen. As a result of oxidation, scale is formed on the tubes, which may be detected by its color, which turns dark blue.

When scale is chipped off the walls of the tubes, they become thinner. Coke forming on these walls greatly reduces their thermal conductivity and prevents the normal heat transfer, so that the outer walls of the tubes must be more strongly heated. As a result of the thinning of a tube from oxidation and overheating,

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caused by increased deposition of coke, it bulges and finally bursts at the thinnest place, or, as they say, burns out. When a tube burns out, liquid under pressure bursts into the furnace chamber and ignites. Flame then bursts out of all parts of the furnace that are not absolutely tight, and thick black smoke issues from the smokestack, and sometimes flame as well. In case a tube burns out threatening accident, the following measures must immediately be taken: (a) stop the hot-oil pump, (b) put out the burners, (c) pass steam into the combustion chamber of the furnace, (d) stop the charging pump, (e) pass steam into the tube system if the steam pressure in the steam system is higher than in the tube system.

Further steps will be taken pursuant to the instructions.

To prevent tubes from burning out, defective tubes must be promptly removed from the furnace. A defective tube may be detected by gauging its diameter. The outside diameter of a normal tube is usually 100 millimeters. A tube must be taken out if its diameter reaches 107 millimeters. The roof tubes burn out most often, since they operate under the most severe conditions.

3. Leakage in the return bends. Fluctuations of temperature in a return bend may lead to the development of a leak and ignition of the escaping product. If the leak is small, it will be enough to pass steam into the return bend and let the plant go on operating normally, but if the leak is large, the plant must be taken off stream.

Sometimes it may be necessary to cement up the cracks in the doors of the return bend box to stop the access of air.

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4. Let us consider the most important cases of trouble in the operation of the evaporator.

To avoid coking of the outlet line of the evaporator, gas oil under pump pressure is regularly pumped through it. If the line cokes up in spite of this, the cracking residue is taken off by the reserve outlet line instead. If both lines become coked up, the plant must be shut down.

Leakage in the evaporator seams may occur. Since the evaporator shell is covered by a thick layer of insulation, a small leak in it will be imperceptible at first. However, after the insulation has been soaked by the hot liquid product, the product will take fire. In case of a small leak, it may be possible to continue operating the plant without shutdown. It is only necessary to remove the insulating layer and apply steam to the place of the leak. During the repair period the leak in the seams must be eliminated.

The level of the cracking residue in the evaporator may unduly rise as a result of inattention, thus disrupting the prescribed operating conditions of the plant. If an attendant is careless, and the outlet line is clogged up by coke and the level in the evaporator rises, cracked residue may be carried over into the rectifying column.

5. The following troubles may occur in the rectifying column: carry-over of the cracking residue from the evaporator into the column will result in darkening of the phlegma, and when this enters the furnace it produces very rapid coking of the tubes. When a carry-over of cracked residue is discovered, the level in the

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evaporator must be lowered at once, the phlegma drawn off from the column and fresh gas oil pumped in until its color is completely light again, while the temperature in the furnace must be lowered 15 or 20 degrees Centigrade (to reduce the deposition of coke). If these measures are unsuccessful and the coking-up of the tubes still continues, the plant must be taken off stream immediately.

Undue rise of the phlegma level is reflected in the delivery of charge stock into the furnace, which likewise leads to coking of the tubes. Fresh gas oil must be immediately pumped into the column in this case and the normal phlegma level in the column must be reestablished.

6. The entrainment of gasoline from the gas separator into the gas line leads to gasoline getting into the burners, in which case heavy black smoke issues from the smokestack, as when a tube burns out, and the outlet temperature of the furnace takes an upward jump. To liquidate this accident the burners must be extinguished, the level lowered in the gas separator, the gas line purged, the furnace steam blown and the burners lit again.

7. An emergency stoppage of the hot-oil pump leads to an emergency shut-down of the whole system. The pump may stop on account of valve breakage, interruption of the steam supply, etc. If a leak is discovered in the stuffing boxes, they must be carefully tightened. If this does not help, the pump must be shut down (together with the whole plant) and the gasket changed. A short shutdown of the pump (3 to 4 hours) on account of the stuffing-box gaskets, results in interrupting the operation of the whole plant for 18 to 20 hours, since putting a cracking plant on stream, or taking it off stream, is a lengthy operation. It is

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therefore entirely natural that it is important to do a good job of repairing the hot-oil pump during the current-repair period and give it as much careful attention as necessary during the course of plant operation.

8. Breaking the protection tube of a thermocouple leads to a shutdown of the whole cracking plant. Breakage of the protection tube of the thermocouple in a furnace is particularly dangerous, since owing to the high pressure in the furnace the product will be forced out violently and will take fire. In this case the plant must be shut down at once, purging steam applied to the site of rupture and the fire company called.

9. Breakage of a reducing valve also leads to the whole plant going off stream.

The cases we have enumerated far from exhaust the list of possible troubles and accidents at cracking plants. We have only touched on the troubles that are most frequently observed.

On interruption of the supply of steam, water, electric power or cracking stock, the same measures must be taken as are recommended for atmospheric plants.

79. The Shutdown of a Cracking Plant. Repair and Cleaning

A normal shutdown of a cracking plant proceeds as follows.

To reduce the viscosity of the cracked residue, the level in the evaporator is lowered by one or two test cocks and the temperature reduced to 380 degrees Centigrade. The liquid is then let out of the evaporator which, together with the cracked-residue line, is flushed out with gas oil.

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The temperature in the furnaces is gradually lowered by shifting the indicator of the automatic controller that regulates the temperature of the flue gases over the bridge wall, at first by 10 degrees (for instance, from 730 to 720 degrees Centigrade), then by another 10 degrees, and so on, until the temperature over the bridge wall has dropped to 600 degrees Centigrade. From 600 to 450 degrees Centigrade the temperature is lowered manually, and then the burners are extinguished. After the temperature of the product in the furnace has reached 300-350 degrees Centigrade, the hot-oil pump (or the charging pump, for the small furnace) is stopped, and the furnace steam blown at first in the opposite direction to the feed flow and then in the same direction, for 2-3 hours each time.

After the steam blowing of the furnace has been properly adjusted, the purging of the columns is commenced (first draining the liquid out of them). This takes the same amount of time. After the furnace has been blown through the plugs are loosened and then cautiously opened, first in one section and then in another, to make sure that there is no product left in the furnace. The gate valves on all apparatus of the plant are closed, and the lines between them are capped.

Depending on the condition of the equipment, the intermediate repair period of a cracking plant takes from four to six days (and even as long as ten days, if the furnace has been very badly coked up).

During the repair period of a plant the following work is done: (a) the equipment is cleaned of coke, (b) the diameter of the

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furnace tubes is checked and the unsatisfactory tubes replaced; (c) all equipment is gauged and inspected to determine the extent of wear; (d) all pumps are overhauled and defective details and stuffing-box gaskets replaced; (e) all defective packing on the lines and apparatus is replaced and (f) the control instruments are checked and repaired.

Cleaning the tubes in the furnace of a cracking plant is a very time-consuming and laborious task; it is also the main factor determining the length of the repair period. It commences as soon as the plugs are opened while the tubes are still hot. It is done by special pneumatic hammers (Figure 101) with miniature turbines, hammer heads or cutters (Figure 102) with pneumatic drills mounted on the ends. To speed up the cleaning of the tubes, 10-12 men are usually assigned to this operation (Figure 103). The work requires experience, since an unskilled workman may either leave a ring of coke when cleaning it out or damage the inner walls of the tube if the hammer strikes the same place too often.

The coke is burned out of return bends by the flame of an autogenous torch. Upon completion of cleaning, the inside and outside diameter of the tubes is gauged. The inside gauging is done by special instruments at five points in the tubes. The outside diameter is gauged with outside calipers from a stand set up inside the furnace (after it has cooled to 55-60 degrees Centigrade). The results are entered in a journal. As we have already noted, when the outside diameter is enlarged to 107 millimeters the tube should be taken out. After the tubes have been changed (but before the plugs have been closed) all the tubes are inspected to make sure that no foreign object has gotten into them.

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After steam has been blown through the evaporator, water is introduced into it through the upper manhole, so as to cool off the coke quickly, then the lower manhole is opened and the cleaning out of the coke begins. So much coke may accumulate that sometimes it takes up more than half the volume of the evaporator. It is removed by crowbars, which takes a good deal of time (2 - 5 days).

The cracking-residue line and the tubes of the cracking-residue cooler are taken apart and the coke removed in the same way as from the furnace tubes.

In the columns the hatches are opened, the condition of the plates and bubble caps checked and the unsuitable ones replaced; the sediment and coke is removed from the plates.

80. Starting Up a Cracking Plant after Repairs

The starting up of a cracking plant consists of the following operations: (a) inspection of the equipment and filling it with gas oil, pressure testing the equipment, (c) circulation and (d) transition to cracking.

Inspection of the equipment. The tightness of the plugs and hatches and the holding power of the flanges and gate valves is thoroughly tested (by tapping with a hammer). All foreign articles (plates, pieces of tubes, etc) are then removed from the furnace and apparatus. All gate valves in the plant must be closed; they are opened as necessary in filling the equipment with gas oil, on starting up the plant. Gas oil is first supplied to the column (up to the 4th test cock), then to the evaporator, and then to the furnace. In filling the furnace all plugs are tested for

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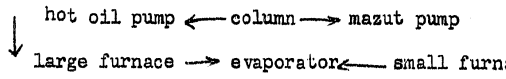
leakage, and any leakage found is immediately corrected. Then all the remaining equipment is filled with gas oil. Steam is blown through the manometer lines. The hot-oil pump is warmed up and tested first empty and then with product.

Pressure testing of the equipment. The furnace is pressure tested two times (in some plants, three times): at 35-40 atmospheres and at 100-110 atmospheres. At 35-40 atmospheres, the pressure test is run with the gate valve to the evaporator closed. The plugs of the furnace and all unions as far as the evaporator are carefully inspected. If leakage is detected anywhere, the pressure in the equipment is reduced and the leakage corrected. The reducing valve is then closed, and the furnace pressure tested under 100-110 atmospheres pressure. If there is no leakage, the furnace is considered ready for operation. The small furnace and the exchangers are tested under 30-35 atmospheres pressure. The column and the evaporator are tested by steam under 6 atmospheres pressure.

Circulation in a Winkler-Koch plant is in two directions: (1) column bottom -- hot-oil pump -- large furnace -- evaporator -- balancing line -- column; (2) column -- mazut pump -- exchanger -- small furnace -- evaporator -- balancing line -- column.

The flow in such a plant may be schematically represented as follows:



In the Nefteproyekt system two-coil plant the flow is as follows:

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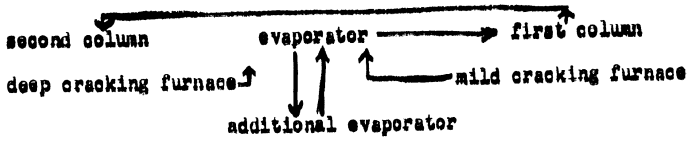

When the circulation has been adjusted, the firebox of the furnace is blown out with steam and two or three burners are turned on. After 30 minutes this number is increased to four or five. The temperature of the circulating gas oil must be raised at the rate of 50 degrees an hour. The water in the equipment is carried along by the gas oil, accumulated in the evaporator, and is then drained off by the lower drainage line. Some of the water is evaporated and removed through the steam blowing valve (at the top of the evaporator). When the temperature of the product in the evaporator reaches 150 degrees Centigrade the valve is closed. When the temperature of the gas oil is increased to 200 degrees Centigrade and the lighter fractions begin to evaporate, the gate valve from the condenser to the gas separator is opened. When the outlet temperature of the product from the furnace reaches 425-430 degrees Centigrade a mild cracking of the feed stock occurs forming gas and gasoline. When the temperature of the product in the furnace is 460-470 degrees Centigrade and 360-370 degrees Centigrade in the evaporator, the cracking becomes intensified and the plant, as they say, is shifted to cracking. For this purpose the circulation of the product from the evaporator to the column is stopped, the plant is switched over to mazut, and the gate valves on the route of the cracking residue from the evaporator into storage are opened. The level of the liquid in the evaporator and the columns is now established between the second and third test cocks.

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When a temperature of 160 degrees Centigrade has been reached at the top of the second column, the delivery of reflux by the reflux pump is adjusted. As the pressure in the gas separator rises to 0.5 - 0.7 atmosphere, burner after burner is gradually shifted to liquid fuel, care being taken to prevent any sudden jumps in temperature. The operating conditions of the plant must then be regulated in accordance with the prescribed process chart on the basis of the laboratory data. The rest of the process will follow the procedure already outlined above.

[Pages 190 to 200 omitted by request]

As already noted, the yield of coke in thermal cracking is limited to 0.1 percent of the residue, while in catalytic cracking it may reach 4 - 5 percent.

In thermal cracking, reactions of decomposition readily take place, as a result of which a considerable amount (10 - 20 percent) of gas rich in the light hydrocarbons (methane, ethane, ethylene) is formed; in catalytic cracking, the yield of gas is lower (5 percent), and it is rich in the less valuable hydrocarbons (propylene, isobutane, isobutylene). In thermal cracking condensation reactions of the unsaturated hydrocarbons take place just as readily, forming a large quantity of condensation products in the form of a heavy high-viscosity cracked residue; in catalytic cracking almost no condensation products are formed, and no cracked residue at all is produced.

Thus, the principal reactions of thermal cracking are the decomposition of hydrocarbons (with the paraffin hydrocarbons being

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the easiest to decompose) and the condensation of the unsaturated hydrocarbons (with formation of high-molecular products). The cyclic hydrocarbons (naphthene and aromatic) keep their cyclic structure as a rule, but the side chains are split off and gases formed.

In catalytic cracking, isomerization, hydrogenation and dehydrogenation reactions mainly take place, together with the formation of carbonaceous deposits. In contrast to thermal cracking, the paraffin hydrocarbons are the worst feed for catalytic cracking, while the reactions with the unsaturated and aromatic hydrocarbons take place most easily of all.

Four products are formed as a result of thermal cracking: (1) a gas rich in the lightest hydrocarbons, (2) a gasoline containing a considerable amount of unsaturated hydrocarbons (30 percent), (3) cracking phlegma, with very high contents of unsaturated and aromatized hydrocarbons and (4) cracked fuel oil.

TABLE 34

MATERIAL BALANCE OF THERMAL AND CATALYTIC CRACKING OF GAS OIL

	Thermal Cracking (with recycling)	Catalytic Cracking (without recycling)
Temperature, degrees C	500	450
Pressure, atmospheres	40	2 - 3
Furnace charge, % (of feed)	300	100
Gasoline, % (of feed)	65	40
Cracked fuel oil, % (of feed)	20	--
Cracked gas oil, % (of feed)		51
Coke, %(of feed)	hundredths	4
Yield of gasoline per pass	22	40

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Three products are formed as a result of catalytic cracking: (1) a gas, rich in valuable hydrocarbons; (2) gasoline with a low unsaturated hydrocarbon content (5-10 percent); (3) catalytic gas oil, rich in paraffin hydrocarbons.

Since the paraffin hydrocarbons are cracked slowly in catalytic cracking, the repeated cracking of catalytic gas oil would be disadvantageous; on the contrary, thermal cracking is only advantageous with recycling.

Table 34 gives a comparative material balance of the thermal cracking of gas oil (with recycling) and the catalytic cracking with a static catalyst (without recycling).

85. The Catalytic Cracking Plant Using a Static Catalyst

A cracking plant using a static catalyst consists of the following principal process units: a pipestill, tar separator, three or six catalyst cases (in the latter case they operate in pairs) with automatic change-over, a rectification column and a gas separator.

Three independent streams flow through such a plant: (1) the product stream, (2) a stream of mixed molten salts and (3) a stream of air to restore the activity of the catalyst (regenerate it).

Let us consider the operation of one of these plants.

The Product Flow (Figure 109)

The feed is drawn from its tank by a pump and pumped through a series of exchangers where it is preheated to about 250 degrees C,

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after which it is delivered to the furnace in two streams. The furnace has two sides, each heated by 28 burners, with a convection chamber in the center. The charge, preheated to 440-450 degrees C, enters the tar separator, which has three bubble plates, refluxed overhead by fresh feed to maintain the necessary temperature. The quantity of vapor passed through the top of the tar-separator is determined by the temperature there. These vapors are then returned to the furnace, into its second half, where they are heated to 470 degrees Centigrade. They then enter the distribution piping system of the catalytic chambers through 24-inch pipes, and are admitted by automatic (motor-actuated) valves to the one of the three pairs of catalytic cases that is on stream at the moment. They pass through the cases from bottom to top through a static layer of catalyst. The temperature in the cases is held at 482 degrees Centigrade and the pressure at 2.1 atmospheres.

A catalytic case consists of a column 11.5 meters high and 3.22 meters in diameter, with a series of tubes through which the oil vapors (during the cracking periods) and air (during the catalyst regeneration periods) are alternately passed. Another series of tubes leads off (alternately) the cracking products and the combustion products (during the period of catalyst regeneration). A third series of tubes form a coil through which molten salts are passed to maintain the necessary temperature in the chamber.

Figure 110 shows the arrangement of a case. The six plates in the case are charged with catalyst. There is a coil with an independent inlet and outlet under each. The feed may be introduced at various levels of the chamber, depending on the desired duration of the contact between feed and catalyst. Figure 111

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shows vertical and horizontal sections of the chamber. It may be seen from the vertical section that the catalyst 1 fills all the space between the molten-salt tubes 2. The horizontal section shows that the tubes are gilled to facilitate dissipation of the heat. The outer walls of the case are covered with a layer of heat-insulating material.

The catalytic cases operate intermittently, each pair being on stream for 10 minutes, during which it is cracking, 10 minutes on catalyst regeneration and 10 minutes on intermediate operations (steam purging, cutting in, cutting out, etc).

Let us analyze the diagram of the operation of three pairs of catalytic cases (Figure 112). While cases 1 and 2 are on stream, cases 3 and 4 are regenerating catalyst for 5 minutes and steam blowing the catalyst for 5 minutes; cases 5 and 6 are being steam blown for 5 minutes after cracking and are on catalyst regeneration for 5 minutes. After these 10 minutes have elapsed, cases 1 and 2 are steam blown for 5 minutes after cracking, and then spend 10 minutes on catalyst regeneration; during this period cases 3 and 4 crack for 10 minutes, and are steam blown for 5 minutes after cracking; in cases 5 and 6 the catalyst regeneration is completed, after which they are steam blown for 5 minutes and crack for 5 minutes. During the following 5 minutes cases 1 and 2 are steam blown and then put on stream for cracking. During this period cases 3 and 4 are regenerated, while cases 5 and 6 complete their cracking operation.

We have traced the operation for the cases for 30 minutes, after which the cycle repeats in the same sequence. At each moment one pair of cases is on stream and cracking, one pair is on catalyst

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regeneration, and the third is on steam blowing. This system assures continuous operation of the cracking plant as a whole, although the cases themselves operate intermittently.

In the control room the cases are automatically switched from one operation to another. The apparatus for the automatic change-over is very complex, and plays a very important part in the cracking process. For instance, the ring-type switch has over 900 contacts arranged in five concentric circles. Special contact rings slide along these contact points. There is a switching impulse every two seconds, which is transmitted to motors actuating the gate valves through a special device. In case of damage to the automatic control, the gate valves may be operated manually.

Besides the ring-type switch there is also a longitudinal-type switch which differs from the former mainly in the arrangement of the contacts.

Let us return to the description of the flow diagram of a static-catalyst catalytic cracking plant. The product vapors leaving the chamber pass through vapor heat exchangers and, after being heated to 390 degrees Centigrade, enter the main rectifying column. Gas oil is drawn from the bottom of the column and passed through a stripping tower (not shown on the diagram) after which they are routed through a cooler into storage. Gasoline is taken from the top of the column and routed through a gas separator and stabilizer into a treatment unit. The characteristics of the gasoline are regulated automatically by the delivery of overhead reflux.

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The Air Flow for Catalyst Regeneration

Air is drawn from the atmosphere through a 30-inch pipe and delivered by a turbocompressor to the air preheater, in which it is preheated by molten salts, and then routed to the catalytic chambers, where the carbonaceous deposits are burned away and a large amount of heat is liberated. The products of combustion of these deposits, containing carbon dioxide, carbon monoxide and hydrocarbon vapors, emerge from the chamber. The carbon monoxide (a product of incomplete combustion) and the hydrocarbons must be subjected to a further process of combustion to convert them into CO_2 and H_2O. The combustion gases are therefore delivered by a 24-inch line to a combustion chamber, in which their combustion is completed by means of a special catalyst. From this chamber the combustion gases are conducted to a gas turbine and actuate it. The turbine in turn drives an air compressor and a 600-kilowatt generator, on which the turbogenerator operates.

The Molten Salt Flow

The cracking reaction absorbs heat, therefore heat must be brought into the reactor to maintain the necessary temperature. This is achieved by circulating a special mixture of hot molten salts (potassium and sodium nitrates) through a pipe coil. A large amount of heat is liberated on regeneration of the catalyst so that its temperature rises. It is not allowed to rise beyond 650 degrees Centigrade, however, or the activity of the catalyst will be impaired. The excess heat must therefore be removed from the case. This is accomplished by circulating the cooled molten salts between the case and an exchanger.

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The molten salts consist of a specially selected mixture of nitrates with a melting point of 149 degrees Centigrade. As a precaution, their temperature is not allowed to drop below 188 degrees Centigrade. The mixture of salts is stored in an insulated tank heated by steam. The total amount of such salts circulating in the system is 453 tons.

The flow of the salts take the following path. They are delivered through a 16-inch line through the bottom of the catalytic cases into the coil. They leave the cases through eight 6-inch pipes and pass into a closed tank. From this tank they flow by gravity through a 24-inch line into the feed heat exchangers to preheat the feed, and then into a special unit to produce steam under 31.5 atmospheres pressure, after which, cooled, they return to the circulation system.

The steam plant consists of a salt-water shell and tube heat exchanger and the auxiliary equipment. Up to 70 percent of the heat liberated on burning out the coke is utilized in this plant.

The static-catalyst catalytic cracking plant is attended by 8 operators and 2 fitters on each shift.

Its fuel consumption is 33 percent higher (per unit of gasoline produced) than that of a thermal cracking plant, but the consumption of steam, water and electric power is lower, amounting respectively to 22 percent, eighty-seven percent and 99.5 percent of that for thermal cracking.

86. Conditions Affecting the Catalytic Cracking Process

The results of catalytic cracking at a static-catalyst

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plant depend on the following basic conditions: (a) characteristics of the feed, (b) temperatures and pressures, (c) contact time, (d) catalyst activity.

The cracking plant described above can be used to process feed of very varied composition, ranging from gasolines (except straight-run gasolines) to mazut and crude. On catalytic cracking of any oil product, motor gasoline of end point 225 degrees Centigrade and octane number 77 - 81 may be obtained. Aviation fuel instead of automobile fuel may be produced by special processing at this plant.

The effect of the chemical composition of the feed. In contrast to thermal cracking, the paraffin hydrocarbons are harder to crack catalytically than the others, while the cyclic and unsaturated hydrocarbons enter more readily into reaction. The principal reactions in catalytic cracking are those of isomerization, dehydrogenation and hydrogenation, and also bursting the rings of the cyclic hydrocarbons, producing isoparaffin or isoolefin hydrocarbons.

(a) Polycyclic aromatic hydrocarbons mainly form carbonaceous deposits in the catalyst. When the feed has a high content of these hydrocarbons, the catalyst is very rapidly poisoned, i.e., clogged up by coke deposits.

(b) Naphthenic hydrocarbons are cracked, opening the ring and forming isoparaffin hydrocarbons. Part of the naphthenes isomerize, changing their rings (six-member to five-member rings and vice versa), and another part is converted to aromatic hydrocarbons (dehydrogenation).

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(c) Derivatives of benzene are cracked, breaking the ring and forming, for example, benzene and an olefin hydrocarbon.

(d) Olefin hydrocarbons are split into lower hydrocarbons, mainly C_4H_8, and are isomerized and hydrogenated on account of the redistribution of the hydrogen. The gasolines therefore contain only an insignificant amount of unsaturated hydrocarbons.

(e) The paraffin hydrocarbons are cracked last of all. The high-boiling paraffin hydrocarbons first give up a molecule of hydrogen or split, forming high-boiling olefin hydrocarbons which then react as indicated under d.

Temperature and pressure. The result of catalytic cracking depends very greatly on the temperature. At temperatures up to 300 degrees Centigrade, the paraffin, naphthenic and cyclic hydrocarbons are almost unaffected, while the olefin hydrocarbons are converted, as a result of isomerization and redistribution of hydrogen atoms, into naphthenes and paraffins.

At temperatures between 350 and 450 degrees Centigrade, the naphthenic and aromatic hydrocarbons, as well as the paraffins, are split. The reactions of hydrogen atom redistribution, isomerization, proceed vigorously. At temperatures over 450 degrees C, decomposition reactions are the principal ones. Therefore, the unsaturated content of gasolines increases when the cracking temperature is raised. The temperature in catalytic cracking varies from 316 to 595 degrees Centigrade, depending on the character of the feed and the desired characteristics of the products. Temperatures of 460-470 degrees Centigrade are employed in cracking gas oil. The pressure is held between 2 and 3 atmospheres.

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Reaction time. The reaction time also depends on the character of the feed. It may be expressed in the form of the ratio between the volume (or weight) of the oil product passing through the catalyst per hour and the volume (or weight) of the catalyst. In the case of gas-oil cracking this ratio is about 2:1.

Catalyst activity. The catalyst used in catalytic cracking plants has a base of aluminum hydrosilicate, which is specially processed and enriched by the addition of the oxides of nickel, cobalt, copper, manganese and other metals.

The composition of the catalyst closely resembles that of activated clays, as may be seen from the following comparative data (in percent):

TABLE 35

	Silicon Oxide	Aluminum Oxide	Iron Oxide	Calcium Oxide	Magnesium Oxide	Other matter
Catalyst	76	16.8	1.6	2.4	1.3	1.9
Gumbrin	66	14.8	2.85	1.71	1.92	0.58

According to experimental data, clay properly processed exhibits the same activity as the catalyst does. The catalyst is prepared in the form of tablets or pellets 4 to 5 millimeters in diameter.

Besides having a high activity the catalyst must also have high mechanical rigidity otherwise it will be rapidly used up by friction, and it must not contain substances that melt at the temperatures used in regenerating.

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As we have already learned, the catalyst becomes covered with coke during the process of catalysis. After regeneration its activity is fully restored. Experience has shown that spent catalyst can be regenerated a few thousand times without perceptible loss of activity. The minimum useful life of a catalyst is 6 months.

In recent years an artificial catalyst with higher activity than the natural one has been developed. This catalyst makes it possible to produce aviation fuel with higher octane numbers; moreover, it cokes less than the natural catalyst does.

87. The Yield and Characteristics of the Products of Static-Catalyst Catalytic Cracking

The yield of gasoline in catalytic cracking is practically independent of the characteristics of the charge stock. In once-through cracking it makes little difference in the yield of motor fuel (in terms of the quantity of oil passed through the catalytic case) whether the feed is crude, mazut, topped crude or gas oil; in all these cases the yield is about 45 percent.

As will be noted from Table 36, 64.6 percent of the crude petroleum is vaporized, leaving 35.4 percent in the tar separator. Of the cracking stock passed through the chamber (64.6 percent of the crude), 3.8 percent of gas, 4.5 percent of coke, 44.5 percent of gasoline and 54.7 percent of catalytic gas oil is produced.

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TABLE 36

RESULTS OF CRACKING VARIOUS CRACKING STOCKS BY A CATALYTIC CRACKING PLANT WITH FIXED CATALYST

	Crude Oil	Topped Crude	Light Mazut	Heavy Mazut	Heavy Gas Oil	Light Gas Oil
Yield of cracking stock, % of crude	100	66.7	52.4	38	--	--
Residue on evaporation, % of crude by volume	35.4	8.4	8.4	8.0	--	--
Charge to chamber, % of crude by volume	64.6	58.3	44.0	30.0	--	--
Dry gas, % by volume	3.8	4.0	5.4	5.1	4.9	4.3
Coke in % of charge to case	4.5	3.5	5.3	5.4	1.0	3.3
Cracked gasoline, % by volume	44.5	41.9	44.1	42.6	50.3	44.7
End point of gasoline, degrees C	202	227	212	211	210	203
Catalytic gas oil, % by volume	54.7	57.7	51.7	56.4	49.9	53.0
Specific weight of gas oil	0.917	0.872	0.884	0.893	0.895	0.849

The crude yielded 38 percent of mazut, of which 30 percent was vaporized and entered the catalytic case, while 8 percent remained in the tar separator.

The feed entering the catalytic case produced 5.1 percent gas, 5.4 percent coke, 42.6 percent gasoline and 56.4 percent catalytic gas oil.

Thus, the general conclusion may be drawn that in once-through catalytic cracking:

The yield of motor fuel does not depend on the properties of the feed and is roughly equal to 45 percent (by volume),

the yield of gas does not exceed 5 - 6 percent,

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the total yield of liquid products is high,

over 50 percent of catalytic gas oil is produced and may be recracked in catalytic or thermal-cracking plants.

One of the most important achievements of the catalytic cracking process with static catalyst is the possibility of producing high-grade aviation fuel. The following products are obtained in producing this fuel:

Gas	13-15 % (by weight)
Coke	2-6 % (by weight)
Aviation fuel	35-53 % (by volume)
Ligroin	10-20 % (by volume)
Gas oil	35-50 % (by volume)

The characteristics of the products. Regardless of the properties of the feed used, the characteristics of the motor fuel remain approximately the same: it is distinguished by high stability, low gum and low sulfur, and its octane number ranges from 77 - 80.

The grow chemical composition of catalytic gasoline differs considerably from that of thermally cracked gasoline (Table 37).

TABLE 37

GROUP CHEMICAL COMPOSITION OF GASOLINE

Feed Material	Form of Cracking	Paraffins	Naphthenes	Olefins	Aromatics
Ligroin	Catalytic	43	24	5	28
Gas oil	Catalytic	56	19	9	16
Gas oil	Thermal	53	14	30	3
Heavy crude	Catalytic	39	19	9	13

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Catalytically cracked gasolines are rich in isoparaffin hydrocarbons, aromatics and naphthenes.

Under all circumstances catalytically cracked aviation fuels must be recracked in the catalytic chamber, which considerably improves its characteristics. For instance, a gasoline produced from gas oil has the specific gravity 0.727 and the octane number 75.8, but after catalytic processing (refining) its specific gravity is raised to 0.753 and its octane number to 81.9. After addition of 4 cubic centimeters of ethyl fluid per liter, the octane number of this aviation fuel is brought up to 98.

The characteristics of aviation fuel, like those of motor gasoline, are almost independent of the feed stock. For example, the aviation fuel obtained from ligroin has the specific gravity 0.758 and the octane number 98.1 with 4 cubic centimeters of ethyl fluid, while that obtained from heavy gas oil has specific gravity 0.743 and octane number 98.5.

Catalytic gas oil is rich in paraffin hydrocarbons. It may be used as cracking material (as boiler fuel) and Diesel fuel may be obtained from it.

The specific gravity of the catalytic cracking gases ranges from 1.2 to 1.6. Over 50 percent of the gases consist of the butane-butylene fraction.

Tar, the residue from the evaporation of the crudes and mazuts to be cracked, is used as a boiler fuel after mixing with less viscous products, or after mild cracking and as a raw material for producing bitumen or coke.

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Besides this, a fuel with viscosity $E_{50} = 4.8$ may be obtained after mild visbreaking cracking from a tar of viscosity $E_{100} = 22$.

88. The Catalytic Cracking Plant with Powdered Catalyst

The role of the catalytic process with static catalyst is very great. This was the first process to employ a catalyst with good results in the cracking of petroleum stocks. But the technological details of this process are so complicated that soon after the construction of such plants specialists proceeded to develop simpler catalytic plants. As a result of the investigations of the technical world, the plant using a mobile powdered catalyst came into being.

Cracking of the cracking stock takes place in the catalytic cracking plant to be described in the presence of a finely powdered catalyst, a mixture of the charge and catalyst being introduced into the reactor, after which the catalyst is separated from the reaction products, regenerated and again routed to the plant.

The catalyst used in the process is so finely divided that it can be poured like a liquid from vessel to vessel, can be delivered by pumps through piping systems, and its level in the apparatus can be regulated by means of gate valves and valves.

The principal equipment of the plant consists of a feed preheater, reactor, catalyst regenerator, rectifying column, exchangers, steam boiler, air preheater and an apparatus to separate the catalyst from the vapors and gases.

Most of the preheating of the charge is done in the preheater, and therefore the furnace is not so essential an apparatus here as

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in straight-run units. The furnace is utilized only to render the cracking plant more flexible operationally.

The flow chart (Figure 114). The charge, heated in the exchangers 1 and the furnace 2, is delivered to the reactor 5 through the injector 3, which is located in the inlet line of the reactor. In the injector the heated charge is mixed with the hot catalyst, the stream of which is controlled by a gate valve. The mixture of catalyst and charge vapors enters the reactor through the distributor 4. In the reactor it is thoroughly mixed. The vapors and gases then pass through a cyclone separator at the top of the reactor where the catalyst is separated. The catalyst is collected at the bottom of the reactor, purged by steam to remove the hydrocarbons, and routed to the regenerator 12 to restore its activity. The reactor consists of an insulated unit up to 18 meters high and 3.3 to 8 meters in diameter (according to the throughput of the plant). The cyclone separator 6 is mounted in the upper section of the reactor, with a section beneath it in which the spent catalyst is steam blown to eliminate the hydrocarbons in it.

The regenerator consists of a metal vessel lined with firebrick and insulated on the inside. At the top of the vessel, as in the reactor, there is a cyclone separator 14 to remove the catalyst from the products of combustion of the coke. The lower part is equipped with the stripping section 15 in which the catalyst is steam blown. From here it flows by its own gravity into the injector.

The temperature in the regenerator must not go above 560-600 degrees Centigrade. To maintain this temperature, there is forced

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circulation of part of the catalyst from the regenerator into the cooler 18 and back again.

The flue gas from the regenerator enters the steam boiler 21 where the excess heat of the gas is used to generate steam.

The steam boiler is a tubular apparatus in which the water flows through the tubes, while the hot gases pass through the shell side. The gases, cooled to 260-310 degrees Centigrade, are now delivered to the electrical apparatus 22 and pass between the electrodes. The particles of catalyst are collected on the electrodes and are then thrown off into a bunker, while the gases are discharged into the open air. The catalyst is delivered under pressure from the bunker to the regenerator. The regenerated catalyst contains not more than 0.3 - 1.0 percent coke.

Let us return to the description of the flow chart. The cracking product vapors are routed for fractionation to the tower 19. This tower differs from the ordinary bubble towers only in the arrangement of its lower section, in which, under the usual bubble-cap plates there are special settling (contact) plates for washing away the catalyst brought down. The product, drawn from the bottom of the tower, circulates through these plates, washes the finely divided catalyst particles free of the gases and vapors and then flows back to the bottom of the tower. The catalyst is now completely washed free of the gases and vapors and does not spout onto the upper plates. Charge material is preheated in exchangers using the heat from the circulating remainder of the catalyst.

Part of this remainder (3 - 10 percent) is drawn from the tower and delivered to the reactor to replace the catalyst carried over to the tower.

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The gasoline vapors and gas are taken off overhead from the tower, while catalytic gas oil is removed by a side draw.

The normal operation of the cracking plant with powdered catalyst, just as in the static-catalyst plant, is determined by the following conditions: (1) temperature, (2) reaction time, (3) catalyst activity.

The control of the former is considerably simpler than that of the latter.

The principal operating control on the process conditions of the plant is effected by means of controllers. One of them controls the stream of spent catalyst. By moving the control point on this instrument the proportion of catalyst to charge may be adjusted. Another controller regulates the level of the catalyst in the reactor by opening or closing a valve in the line from the regenerator to the reactor. A third instrument regulates the return of the catalyst to the regenerator.

The operation of the plant is very flexible. The characteristics and yields of the products are much the same as those in a static-catalyst plant.

A two-stage catalytic plant is used to produce aviation fuel and differs from the ordinary one only in having two reactors, one of them serving to crack the charge material and the other for the catalytic refining of raw aviation fuel. The two reactors are served by a single regenerator.

89. The Catalytic Cracking Plant with a Mobile Catalyst

Catalytic cracking plants with a mobile catalyst, like those

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with a powdered catalyst, operate, as the name indicates, with a mobile catalyst. The difference between these two processes is that the cracking in the former case takes place in the presence of catalysts in pellet and pill form which are moved by mechanical means, while in the latter case it occurs in the presence of a powdered catalyst that is displaced like a liquid.

The cracking plant consists of the following principal apparatus: a furnace, an evaporator, a reactor, a regenerator (Figure 118), elevators for lifting the spent and regenerated catalyst, a steam boiler to generate steam, cyclone separators, a fractionating tower and a gas separator. The clay-burning kiln used in lube-oil refining plants was also employed on this plant (Figures 115 and 116).

The flow chart (Figure 117). The charge is drawn from the tank by a centrifugal pump and pumped through the product heat exchangers 1, in which it is heated to 260 degrees Centigrade and then delivered to the convection tubes and some of the radiant tubes of the pipestill 12. It then passes, heated to 450 degrees Centigrade, to the evaporator 3 for separation of the heavy tarry residue. The distillate vapors are routed from the top of the evaporator into the radiant tubes of the second half of the pipestill for preheating to the required temperature (480 degrees Centigrade), and then to the reactor 5. The charge vapors move upwards from the bottom of the reactor, countercurrent to the catalyst stream moving down from its top. From the reactor the reaction products are routed to the fractionating tower 8.

Vertical steel reactors (Figure 118) are used in plants of this system. The regenerated catalyst enters at the top, while

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the spent catalyst is drawn out at the bottom of the reactor.

The oil vapors, on the other hand, enter at the bottom, while the reaction products emerge from the top of the reactor. Here, as we see, the principle of countercurrent flow is followed: the fresh and most active catalyst flows countercurrent to the already reacted charge, while the catalyst with weakened activity flows countercurrent to the fresh charge which readily reacts.

In the reactor of a plant with a throughput of 1,5000 tons of charge material a day about 100 tons of catalyst circulate. The reactor has four process zones. (Figure 118).

The first zone is located below the charge inlet. In this zone the spent catalyst is steam blown to remove the hydrocarbon vapors carried over by the catalyst. In the second zone, located above the first one and filled with a layer of catalyst, the process of cracking the charge takes place. In this zone the depth of the catalyst layer ranges from 2.5 to 10 meters according to the throughput of the plant. The catalyst continuously flows in through pipes from the hopper located on the upper level of the reactor down to the second zone. The space between the catalyst level and the inner bottom of the reactor forms the third zone, in which the mixture of hydrocarbons and steam is freed from the catalyst fines carried over by the vapors from the second zone. At the top of the reactor, between the external and internal bottoms, is the fourth zone, filled with regenerated catalyst (from the hopper) and with flue gases delivered with the specific purpose of creating a gaseous cushion to prevent the reaction products from seeping through into the hopper.

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Thus, the reaction section of the apparatus, the second zone, is located between two gaseous layers which prevents the hydrocarbon vapors from seeping into the evaporator or the hopper.

In the operation of a reactor with a constant throughput the following conditions must be carefully observed if gasoline of the required characteristics is to be produced: temperature of the catalyst entering the reactor, temperature of the charge entering the reactor, rate of delivery of the catalyst.

The catalyst used in a plant may be natural or synthetic. The natural catalyst is prepared from special grades of clays by chemical treatment, and consists of irregularly shaped particles with sizes up to 6 millimeters. However, the natural catalyst is not distinguished by high activity and does not possess sufficient mechanical rigidity. The synthetic silicate-alumina catalyst (resembling glass) is manufactured in the form of beads 4 millimeters in diameter. The activity of this catalyst as well as its mechanical strength is considerably higher than in the natural catalyst.

Both types of catalyst are ordinarily used at a cracking plant with static catalyst. However, in producing aviation fuel the synthetic type is mostly used, since it allows production of a higher grade of aviation fuel with a higher yield than the natural catalyst does.

The activity of a catalyst also depends on the degree of its regeneration. Spent catalyst contains from 2 to 6 percent coke. To regenerate the catalytic activity the coke must be burned away. At plants with a throughput of 1,500 tons a day, as

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much as 50 tons of coke are burned away every day.

The spent catalyst is delivered by a mechanical conveyer (elevator) from the bottom of the reactor to the top of the regenerator 7.

The regenerator (see Figure 118) consists of a vertical metal column with a square cross section covered inside by an insulating layer. In it there are a number of zones (up to ten) of combustion and cooling. Each cooling zone has grooves in parallel rows to collect the products of combustion. Air for combustion is supplied to each zone. The cooling zones consist of coils through which water is pumped.

The particles of spent catalyst are delivered to the regenerator from the top, pass downward through grooves and coils and are gradually regenerated. The total length of the regeneration period is about 80 minutes. The maximum admissible temperature during this time is 650 degrees Centigrade, but the regeneration temperature is usually held within the range of 370-600 degrees Centigrade.

The catalyst leaving the regenerator contains 0.2 to 0.5 degrees coke. The spent catalyst is raised by the elevator to the top of the reactor, while the combustion products from the coke together with the minute catalyst particles entrained enter the cyclone separator (see Figure 118), from which the cleaned flue gases are discharged into the air while the catalyst fines are collected below.

Steam under 13-35 atmospheres pressure is generated by heating the water delivered to the steam boiler utilizer.

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Let us return to the flow sheet we are describing. The cracking products pass from the reactor into the bubble tower, where they are fractionated into heavy-gas oil, light-gas oil and a mixture of gasoline vapors with steam. Part of the heavy-gas oil is mixed with the charge and recycled, part is delivered to the exchanger and the rest is returned to the tower. The gasoline is taken off overhead.

If necessary the gasoline produced may be rerun by catalytic refining. The yield of cracking products depends on the character of the charge and the conditions of process. 35-45 percent gasoline is produced from gas oil by once-through cracking (without recycling), and 50-60 percent if recycling is employed.

Table 38 gives the approximate data of the yields from gas-oil cracking.

TABLE 38

YIELD OF PRODUCTS FROM THE CRACKING OF GAS OIL

	Without recycling	With recycling
Temperature in degrees C	455	460
Pressure in atmospheres	0.7	0.7
Yield of motor gasoline in % by volume of crude	39	56
Catalytic gas oil, %	48	26
Butane-butylene fraction, %	13	15
Gas, %	5	5

The characteristics of the motor fuel vary somewhat depending on the character of the charge (Table 39).

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TABLE 39

CHARACTERISTICS OF MOTOR FUEL

	From paraffin-base gas oil	From mixed-base gas oil	From naphthene-base gas oil
Octane number, clear	76.9	78.1	82
Octane number, +3 cc	95.1	96	99.2
End point, degrees C	207	208	210

When aviation fuel is produced by the plant its yield is 20-23 percent, with 8-10 percent ligroin and 50-56 gas oil.

This fuel contains a high percentage of aromatic hydrocarbons.

The process may be regulated in such a way as to yield a fuel with 75 percent of the aromatic hydrocarbons benzene, toluene and xylene.

Advantages of the mobile-catalyst cracking plant as compared to the static-catalyst plant.

1. Continuous operation of regenerator and reactor.
2. Instead of six catalytic cases the plant has two units, a reactor and a regenerator.
3. The system of controlling the equipment is simplified.
4. Water may be used instead of molten salts.
5. Instead of turbines and an air compressor, a single air blower is sufficient.

In spite of the fact that the catalyst consumption is considerably lower at the static-catalyst plants than at mobile-catalyst plants (0.02-0.03 percent against 0.1-0.3 percent per ton of throughput), the former are being displaced by the latter in consequence of their simplicity and ease of service.

Figure 119 shows a general view of a mobile-catalyst cracking plant.

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CHAPTER NINE

GAS RECOVERY AND GAS FRACTIONATION

90. Natural and Artificial Petroleum Gases

Gas is almost a necessary companion of petroleum. But rich deposits of oil gas not connected with petroleum are also encountered, for instance, the Saratov gas field.

The mixture of hydrocarbons separated from petroleum is termed natural petroleum gas, while gas produced from deposits of gas alone is termed natural gas.

Natural petroleum gases may be dry or poor, or they may be rich. The dry (poor) gases consist of 75-98 percent methane and are almost free of the gasoline hydrocarbons, they are termed dry. The wet (rich) gases consist of 35-50 percent methane, 7-20 percent ethane, 10-25 percent propane, and they can contain as much as 10 percent light natural gasoline.

TABLE 40

COMPOSITION OF NATURAL PETROLEUM GASES (IN MOL PERCENT)

	SPECIFIC GRAVITY	H_2S	N_2	CO_2	CH_4	C_2H_6	C_3H_8	C_4H_{10}	C_5H_{12}
Kali (Baku)	0.602	--	--	1.6	95.3	2.3	0.5	0.3	--
Novo-Grozniy	1.136	--	--	--	39.7	9.5	19.0	18.4	13.3
Ishimbay	0.888	3.1	1.0	1.5	57.3	2.8	13.2	10	11.1
Maykop (S Ye)	1.560	--	--	4.5	13.8	23.0	32.9	18.8	7.0

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Gases of varied composition are shown in the Table. The first of them at Baku, consists almost entirely of methane (95 percent); the second, Groznyy, contains 13.3 percent natural gasoline and only 39.7 percent methane. The specific gravity of the first is therefore only 0.602, while that of the second is 1.136.

The petroleum gas of the Ishimbay deposit is one of the rich gases (with 11.1 percent natural gasoline) and is distinguished by a very high hydrogen sulfide content (up to 3.1 percent. When natural gasoline is produced from this gas, the gas must first be freed from the hydrogen sulfide.

Maykop gas (Stratum Ye) has a very high specific gravity (1.56) and is distinguished by a low methane content (13.8 percent).

When crude is processed at refineries (for example, in cracking, pyrolysis and other processes) refinery gases, or artificial gases, are produced. These gases are distinguished by high unsaturate content, the gases from vapor-phase cracking and pyrolysis having the highest (put to 45 percent and 30 percent, respectively). The gases from liquid-phase cracking and those from catalytic cracking have about 15 percent (Table 41).

The products obtained from these gases are tremendously important for the national ecenomy, and include stable natural gasoline, separate hydrocarbons that serve as raw material for various industrial processes, dry gas consisting of methane and ethane, liquid gas consisting of propane and the butanes.

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TABLE 41

	CRACKING UNDER PRESSURE	WINKLER-KOCH CRACKING PLANT	VAPOR-PHASE CRACKING	PYROLYSIS	CATALYTIC CRACKING
Hydrogen	3	4.9	9	12.0	5.4
Methane	50	42.3	28	56.8	9.9
Ethane	17	19.5	14	--	3.1
Propane, butane	10	13.4	3	--	24.1
Isobutane	5	5	1	--	42.9
Ethylene	2	3.9	20	16.3	3.0
Propylene	8	6.8	15	9.0	6.1
Butylene	5	3	5	1.6	5.0
Isobutylene		1.2	3.5	2.4	0.5
Divinyl	--	--	1.5	1.1	--

91. The Methods of Extracting Natural Gasoline

The petroleum gases are routed to natural gasoline plants for processing into natural gasoline, liquid and dry gases, and individual hydrocarbons.

Natural gasoline is extracted from gas by three methods: (1) compressor, (2) absorption, (3) adsorption.

THE COMPRESSOR METHOD of extracting gasoline from gas consists in compressing the gas to a certain pressure, cooling it in coolers and then separating the gasoline in separators.

The degree of extraction depends on the temperature and pressure; the lower the temperature and the higher the pressure, the more complete the extraction.

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THE ABSORPTION METHOD is based on the ability of liquids to absorb gases and vapors. It consists of two operations: (1) absorption of the gas of gasoline by an absorbent oil and (2) extraction of the gasoline from the oil at a distillation plant.

The absorption takes place in a specially equipped column -- the absorber (Figure 120). The gas enters at the bottom of the absorber and ascends through it, while the absorbent oil is delivered from the top of the absorber and is drawn off at the bottom. Solar and gas-oil distillates are used as absorbent oils.

The gasoline is extracted from the oil (desorption) in an ordinary rectifying column. After extraction of the gasoline, the absorbent oil is again used for the absorption process.

THE ADSORPTION METHOD is based on the absorption of gases by solids (for example, by activated charcoal or silica-gel). The gases are passed through an adsorber filled with the absorbent. The heaviest hydrocarbons are absorbed by the pores of the solid, while the light hydracarbons pass out of the adsorber. The adsorbent is usually saturated in 0.5 - 1.5 hour, after which the adsorber is taken off stream (and another one substituted), and steam is blown through it. The gasoline vapors pass into the condenser together with the steam. The adsorber is put on stream again after the steam blowing.

92. Gas Fractionators

At compressor and absorption plants dry gas containing methane and ethane is produced, together with raw natural gasoline containing gaseous hydrocarbons: butanes, propane, a small amount

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of ethane and a very small amount of methane. Natural gasoline plants also stabilize gasoline at gas fractionators, which may produce: (1) stabilized gasoline and gas, (2) stabilized gasoline and liquid gas (a mixture of propane and butanes), (3) stabilized gasoline and individual hydrocarbons.

The flow charts of gas fractionators differ among themselves according to the direction of the processing selected.

STABILIZATION OF NATURAL GASOLINE (Figure 121). The principal equipment of the process unit consists of a stabilizer, cooler, exchanger and stripping still. The raw natural gasoline is pumped through the exchanger 2 and the stabilizer 11. The stabilizer consists of a bubble tower with 18 to 36 plates.

The stabilizer is heated by the circulation of gasoline through the steam stripping still 6. The light hydrocarbons stripped from the gasoline ascend through the stabilizer. The gas passes out at its top and then enters the fuel gas line through the cooler 4 and the reflux collector 5. The required composition of the waste gas is maintained by overhead delivery of reflux to the stabilizer.

The stabilizer gasoline is routed to storage through the stripping still 6, the beed-heat exchanger 2 and the cooler 3. The stabilized gasolines produced by this method contain a considerable quantity of butanes.

STABILIZED NATURAL GASOLINE AND SIMULTANEOUS PRODUCTION OF LIQUID GAS. In this case the scheme of the plant will differ from the one just described by the presence of a column for liquefied gas, provided with a stripping still, a condenser and

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other equipment.

The operating conditions for the stabilizer are set up so as to take overhead from the stabilizer a gas mixture that also contains butanes. Part of the gases are condensed and returned as reflux to the stabilizer. The rest of the gases pass through the liquid gas column, in which they are separated into dry gas consisting of methane and ethane, and wet gas, consisting of propane and butanes. The dry gas is taken overhead and the wet gas from the bottom.

STABILIZATION OF NATURAL GASOLINE WITH SIMULTANEOUS PRODUCTION OF INDIVIDUAL HYDROCARBONS. The plant used for this purpose has a number of columns, in each of which as individual hydrocarbon is produced: propane, isobutane, normal butane. The system of the gasoline plant at Ishimbay, which produces individual hydrocarbons, is considered below.

93. The Gasoline Plant at Ishimbay

The plant is designed to produce stabilized gasoline, normal butane, isobutane, propane and dry gas.

The principal equipment consists of two absorbers, a scrubber, a dephlegmator, a stripping column, separators, a propane column, a gasoline stabilizer, a butane column, preheaters and a cooler.

THE FLOW SHEET (Figure 122). From the oilfield compressor stations the gas is delivered, after purification from sulfur compounds, under 5 atmospheres pressure to the absorbers 1,

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which have 20 plates each and operate alternately. The gas enters at the bottom of the absorber, while the absorbent oil, delivered from the tank 7, flows from the top countercurrent to the gas. The dry gas is routed through the scrubber 3 into the distribution system. The saturated absorbent oil passes through the exchanger 3 and the steam preheater 4 into the demethanizer 5, which has 10 plates and operates under 15 atmospheres pressure.

Cold absorbent oil is delivered at the top of the demethanizer, while steam is passed in at the bottom to help in stripping the methane. The dry gas is taken overhead from the demethanizer and passed to the distribution system. The saturated oil flows by gravity into the stripping tower 6, which has 20 plates.

The tower operates under 6 atmospheres pressure, at a bottom temperature of 180 degrees Centigrade and at 105 degrees Centigrade on top. The gasoline vapors are stripped in it from the saturated oil, which then passes to the tank 7 through the exchanger 3. The gasoline vapors leave the stripping tower, are condensed in the condenser 8, enter the separator 9 and then the raw gasoline tank 10. The uncondensable gases are again returned to the absorber.

The following operation peformed at the plant is the stabilization of the gasoline and the production of the individual hydrocarbons.

The raw gasoline is purified to remove the sulfur compounds and is gathered into the feed tank 11 for fractionation. From this tank it is pumped through the preheater 12 and the depropanizer 13,

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which has 30 plates and operates under 22 atmospheres pressure at 168 degrees Centigrade on the bottom and 56 degrees Centigrade overhead. Propane, mixed with fixed gases, leaves the top of the depropanizer. From the separator 9 of the depropanizer, the fixed gases are returned to the absorber, while the liquid propane enters the storage tank for finished propane. The hot gasoline from the depropanizer flows into the receptacle 14, from which it flows by gravity into the stabilizer 15.

The stabilizer has 30 plates and operates under 6-7 atmospheres pressure; the bottom temperature is 140 degrees Centigrade and the overhead temperature 59 degrees Centigrade. The stabilized gasoline flows out of the stabilizer through the cooler 16. The mixture of butanes is taken from the stabilizer top and passes through the condenser and separator into the tank 17. It is then separated in the butane column 18 into normal butane (taken at the bottom) and isobutane (taken overhead). This column has 50 plates and operates under 7 atmospheres pressure at 70 degrees Centigrade at the bottom and 51 degrees Centigrade overhead.

The plant produces the following products:

Stabilized gasoline	35.8 %
Normal butane	8.5 %
Isobutane	4.4 %
Propane	4.4 %
Dry gas	46.4 %
Losses	0.9 %
Total	100.0 %

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94. The Fractionation of Refinery Gases

Not so long ago cracked gases were still used mainly as a fuel. Today these gases are utilized as a valuable raw material in producing a whole list of products, especially high-grade aviation gasoline.

As already remarked, the cracked gas is separated from cracked gasoline in a gas separator. The gasoline is thereby taken off with some content of dissolved gas, while the gas entrains gasoline, the quantity so entrained increasing with the pressure in the separator. For instance, at 3 atmospheres pressure in the separator, the gas may entrain up to 17 percent gasoline hydrocarbons, amounting to 3.5 percent of the gasoline. The gasoline is freed from the gas in the stabilizing plant, operating under the principles above described. The gas is freed from gasoline by the aid of the absorber.

Not all the gas is used in the production of the high-octane components used to produce high-octane aviation fuels, but only the unsaturated hydrocarbons it contains: normal butylene, isobutylene, propylene and some of the ethylene. Isobutane is the paraffin hydrocarbon that is mainly used for this purpose. The cracking gases are therefore fractionated in gas fractionating plants that separate not the pure hydrocarbons but the propane-propylene, butane-butylene and pentane-1-pentene fractions.

A gas-fractionating plant consists of a methane-ethane column, a propane column, a butane column, heat-exchangers, coolers and other equipment.

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The gas is delivered to the methane-ethane column by a compressor. Hydrogen, methane and ethane are taken off at the top of this column. The gas, freed from the light hydrocarbons by the first column, next passes to the second, or propane, column, in which the propane-propane-propylene fraction is separated from the gas. The remainder of the gas enters the third, or butane, column, for separation into a butane-butylene fraction, taken off overhead, and a pentane-pentene fraction, mixed with gasoline residues, taken off at the bottom.

Composition of the propane-propylene fraction:

C_2	up to 2 %
C_3	up to 97 %
C_4	up to 1 %

Composition of the butane-butylene fraction:

C_3	up to 2 %
C_4	up to 97 %
C_5	not over 1 %

97. Gas Scrubbing

The gases produced in processing sour crudes contain up to 4-6 percent sulfur. Hydrogen sulfide and mercaptan are harmful substances with highly corrosive properties. The presence of sulfur compounds in gasoline, moreover, considerably lowers the octane number, since it reduces the lead susceptibility. The gases must therefore be freed from sulfur compounds before being processed. This is done, for instance, by sodium phenolate.

The method of phenolate scrubbing of gas is based on the

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following reaction:

$$C_6H_5ONa + H_2S \rightleftarrows NaHS + C_6H_5OH$$

sodium phenolate + hydrogen sulfide ⇄ sodium hydrosulfide + phenol

The reaction is reversible and at low temperatures proceeds from left to right, while at higher temperatures it proceeds from right to left.

The plant consists of an absorber, desorber, dephlegmator, exchanger and cooler.

The flow sheet (Figure 123). The raw gas enters the bubble-cap absorbing column, or absorber. A cooled solution of sodium phenolate is delivered countercurrent to the gas from a mixer for soda and phenol. The scrubbed gas is taken off overhead from the absorber, while the phenol solution passes through the exchanger and supply tank into the desorber or activator. Since a high temperature is maintained in the desorber, the above reaction proceeds from right to left, and the solution flowing downward through the plates is freed from hydrogen sulfide. The hydrogen sulfide is eliminated from the top of the desorber, while the solution of phenol and caustic soda, freed from hydrogen sulfide, passes out of the bottom into the tank.

In spite of the simplicity of the flow sheet, the process proceeds unevenly. Its principal fault is the entrainment of phenol (from the absorber) by the gas. To reduce this entrainment water is delivered at the top of the absorber.

Up to 98 percent of the hydrogen sulfide is removed from the gas by the phenol scrubbing.

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STAT

CHAPTER TEN

PRODUCTION OF THE COMPONENTS OF HIGH-OCTANE GASOLINES

96. The Significance of High-Octane Aviation Fuels

The development of the production of aviation fuels is not lagging behind the rapidly growing demand of aviation for fuel.

During the last few years alone, the world production of aviation fuel has increased by tens of times. Together with the quantitatively increasing demands of aviation, it also makes high demands with respect to the quality of aviation fuel. The octane number of the fuels used in civil and military aviation (Table 42) is rising from year to year in accordance with these demands.

TABLE 42

Years	Octane numbers of aviation fuels employed	
	in military aviation	in civil aviation
1932	82	73
1934	90	80
1936	93	85
1938	98	89
In the war years	100 and up	--

The ability of the oil industry to produce high-octane aviation fuels from crudes is limited first by the quantity of crude produced, second by the quantity of aviation fuel contained in that crude, and third by the quality of the aviation fuels.

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Many different methods of preparing high-octane aviation fuels have appeared in recent years. One of these methods -- catalytic cracking -- has considerably increased the resources of aviation fuels, but it cannot produce fuels with an octane rating of 100 and upwards.

At the present time the method of blending three components to produce high-octane gasolines has been generally adopted:

(1) the base gasoline or base stock, including straight-run gasolines and catalytic cracked gasolines;

(2) a high-octane component or special blending component, including individual hydrocarbons -- isopentane, isooctane, toluene, isopropylbenzene, aviation alkylate, neohexane -- or close-cut hydrocarbon fractions with an octane number around 90;

(3) ethyl fluid, or tetraethyl lead (TEL).

In this chapter we shall become acquainted with some of the industrial methods used for manufacturing high-octane compnents.

97. The Production of Isooctane by Polymerization

Pure isooctane has an octane number of 100, while for technical isooctane it is from 91 to 95. It has been used as a component of high-octane aviation fuel since 1935.

Two methods are used to produce isooctane.

The first method consists of subjecting the butane-butylene fraction separated at a gas fractionating plant to a process of polymerization (condensation of two or three molecules into one) at a polymerization plant. (Figure 124) Diisobutylene

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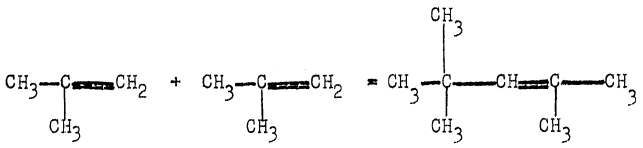
is produced in this way, and is converted into isooctane at a hydrogenation plant.

With the second method, isooctane is produced at once at a single plant by what is termed alkylation.

SULFURIC ACID POLYMERIZATION. Polymerization may take place either at high temperatures, in which case it is termed thermal, or in the presence of catalysts, when it is known as catalytic.

Two substances are industrially used as catalysts, sulfuric acid and phosphoric acid.

To obtain isooctane, two molecules of isobutylene must react together:



If two molecules of normal butylene, or one molecule of normal butylene and one molecule of isobutylene combine, diisobutylene is not formed. It is therefore aimed to choose the conditions (reaction temperature, strength of the sulfuric acid, reaction time) such that the isobutylenes will be the principal components to react. Under treatment with sulfuric acid of 65 degrees Beaume at 20-35 degrees Centigrade, 90 percent of the isobutylene is converted to diisobutylene, while normal butylene hardly reacts under these conditions.

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At 70-90 degrees Centigrade and 70 percent acid strength, normal butylenes also react. Although the octane number of the product is lower than in cold sulfuric acid polymerization, the yields are considerably higher.

POLYMERIZATION BY PHOSPHORIC ACID CATALYZER (Figure 125). The butane-butylene fraction (BBF) is delivered by the compressor 1 to the preheater 2 (preheating is carried to 165-175 degrees Centigrade), and then to the reactors 5, operating under 45-50 atmospheres pressure.

The approximate composition of BBF is: propane-propylene fraction, 1 percent; normal butylenes 33 per cent, isobutylenes 18 percent, butanes 48 percent, After polymerization the isobutylene content falls to 0, and the butylene to 5-10 percent. The greater the proportion of normal butylenes entering the reaction, the lower the octane number of the product. After rectification in the column 7, the diisobutylene has the following properties: initial boiling point 93 degrees Centigrade, end point 131 degrees Centigrade, octane number 86-88.

HYDROGENATION. Diisobutylene contains a single double bond. Hydrogen must be added to it to obtain a finished product. The process of hydrogenation is carried out in reactors operating at about 200 degrees Centigrade, under 300 atmospheres pressure, in the presence of a nickel catalyst.

To carry out hydrogenation at a refinery, the production of catalyst and hydrogen must be organized. The plants that produce isooctane by this method are complicated, bulky and require a considerable number of auxiliary units.

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PRODUCTION OF POLYMER AVIATION FUELS. Polymer aviation fuels may be produced by polymerization of any cracking gas (without fractionating it) on a phosphoric-acid catalyst.

The gas from the gas separator is delivered to a preheater and heated to 180-200 degrees Centigrade, and passed with steam into the reactor. The unreacted gas is separated in the stabilizing column to which the reaction products then pass. Gasoline of the required characteristics is taken off in a rerun column.

The polymerization reaction is conducted in such a manner that 94-100 percent of the isobutylene, 92 percent of the butylene, 90 percent of the propylene and 40 percent of the ethylene react.

The octane number of the polymer gasoline is 82-84 and its specific gravity is 0.710-0.720.

Polymer gasoline may also be produced by thermal polymerization of gas under the following approximate conditions: temperature 510 degrees Centigrade, pressure 55 atmospheres. (Another method used a temperature of 700 degrees Centigrade and 2-3 atmospheres pressure).

The quality of the gasolines produced by thermal polymerization is lower than that of catalytic polymer gasolines.

98. Alkylation

Alkylation means the substitution of a hydrogen atom in an organic compound by one of the groups called alkyls: methyl, CH_3, ethyl, C_2H_5, propyl, C_3H_1, etc.

Alkylation today is the principal process for manufacturing high-octane components for aviation fuel from the gaseous hydrocarbons.

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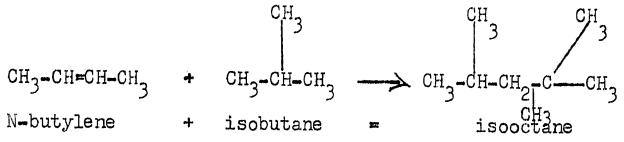
The products of alkylation are used as the main blending additive in the manufacture of any kind of aviation fuel.

Of what does the process of alkylation essentially consist, and why has it been so widely adopted?

In textbooks on organic chemistry we may read that the paraffin hydrocarbons do not enter into combination reactions since they are inert and inactive substances. This is not invariably true. The paraffin hydrocarbons of isomeric structure combine fairly readily even at low temperatures with unsaturated hydrocarbons to form saturated hydrocarbons. For example, a molecule of isooctane is produced from a molecule of butylene and a molecule of isobutane:



On alkylation, saturated hydrocarbons of isomeric structure are produced with high octane numbers and good lead susceptibility.

The alkylation method possesses great advantages over that of polymerizing the paraffin hydrocarbons: it is cheaper and simpler than the two-stage process of polymerization and hydrogenation.

A tremendous advantage of alkylation is also the possibility of expanding the raw material base by using it. In polymerization, isooctane is obtained only from isobutylene and butylene, but in alkylation, for every kilogram of reacting unsaturated hydrocarbon about 1 kilogram of isobutane is utilized besides these.

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For example, BBF contains 20 percent isobutylene and 15 percent butylene. While by polymerization a maximum of 20 kilogram of isooctane can be obtained from 100 kilogram of the raw material (if the isobutylene alone is polymerized), or 35 kilogram of isooctane (if all the butylene is polymerized), by alkylation the 35 kilogram of butylenes may be caused to react, together with about the same quantity of isobutane, giving a yield of 70 kilogram of the finished product. Consequently, twice as much isooctane can be produced by alkylation as by polymerization. In practice the yield of alkylate amounts to 150-170 percent of the olefin.

Two forms of alkylation are known, thermal and catalytic.

Thermal alkylation is effected at high temperatures (500 degrees Centigrade) and high pressures (ranging from 250-350 atmospheres).

Catalytic alkylation, on the contrary, is conducted at low pressures and almost at room temperatures.

The thermal method of alkylation has not been extensively employed on account of the very severe process conditions involved.

Many different substances have been used as catalyzers for the alkylation process, but two are mainly used in the refineries: sulfuric acid and hydrofluoric acid.

In recent years the alkylation of aromatic hydrocarbons has also become one of the most important processes of the petroleum refining industry. This is explained by the high quality of alkylbenzol as a blending component of aviation fuels, as well

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as by the existence of immense benzol resources (from the pyrolysis of crude oil, and from coking). In recent years the production of cumin -- isopropylbenzene -- has commenced. Cucim is obtained from benzol and propylene in the presence of a catalyst -- phosphoric acid on kieselguhr.

99. Sulfuric Acid Alkylation.

Various olefins and paraffins may be used as stock in the sulfuric acid process of alkylation. In practice, however, olefins from the propylenes (C_3) to the octylenes (C_8) are used, as well as the light ends of cracked gasoline, with isooctane and isopentane as the isoparaffins. Ethylene reacts with great difficulty, while the olefins higher than C_8 yield hydrocarbons with boiling points above those allowable for aviation fuels.

The following is the principal equipment used in sulfuric acid alkylation: reactor, cooler, acid settler, preheater, a number of rectifying columns: to remove the propane (depropanizer), to remove the butane (debutanizer), to remove the isobutane (deisobutanizer) and for redistilling the alkylate.

THE FLOW SHEET (Figure 126). Let us consider the operating scheme of one of the sulfuric acid alkylation plants. The butane-butylene fraction and isobutane serve as the feed for this plant. The BBF is mixed in an assigned proportion with isobutane and delivered to a line to which sulfuric acid is also supplied, which mixes with the feed. The entire mixture is then pumped through the cooler 1 into the reactor 2. So that the reaction may proceed vigorously, the feed and the acid must be agitated until a fine emulsion is formed. It is also necessary

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to have the reaction last long enough to cause all the unsaturated hydrocarbons present to react. This is accomplished by circulating the mixture of hydrocarbons and acid from the reactor to the cooler and back by using the circulation pump 15, as well as by proper design of the reactor with this in mind.

Figure 127 shows two types of reactors used in alkylation plants, the tank reactor 1 and the mechanical reactor 4. The former is a hollow vertical cylinder containing a number of tubes equipped with spray heads. The hydrocarbon-acid mixture is pumped through the tubes and sprayed through the spray heads into the reactor. Thereby a fine emulsion of feed and acid is formed. The mixture is collected by a pump, delivered to the cooler and again to the reactor tubes. The mixture is pumped through a cooler because the heat liberated by the alkylation heats the mixture, and it must be cooled off.

The hydrocarbon-acid mixture is agitated inside the reactor with a mixer. The mixture is cooled by circulating water, passed through a tubular condenser installed inside the reactor. The agitation inside such a reactor is excellent.

The hydrocarbon-acid mixture passes after reaction into the acid settler 3, where it is separated by gravity into two layers: one of acid containing a small amount of organic substances and the other of hydrocarbons.

The spent acid (85-87 percent H_2SO_4) is discharged into the spent acid receiver. The reaction products are routed into the alkali settling tank 4 for neutralization of the sulfuric acid carried over. The spent alkali is then discharged into the spent

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alkali receiver, while the neutralized reaction products are passed through the preheater 5 into the debutanizer 6 for removal of the propane, butane and isobutane from the alkylate, which then, freed of these gases, enters the rectifying column 12 for separation into standard product and a residue of heavy alkylate.

The alkylate is preheated by steam in the preheaters 14. The gases from the debutanizer are passed into the depropanizer 8, from which the propane is taken off overhead and the mixture of butanes from the bottom. The isobutane is then taken off overhead from the deisobutanizer 10 and the butane is taken from the bottom.

The isobutane is again recycled to the system.

CONDITIONS OF SULFURIC ACID ALKYLATION. The ratio between isoparaffin and olefins. The principal reaction that should take place at an alkylation plant is the combination of a molecule of isobutane with each molecule of olefin to produce a molecule of satured hydrocarbon. However, as is commonly known, the unsaturated hydrocarbons easily enter into polymerization reactions in the presence of sulfuric acid, thus producing a single molecule of unsaturated hydrocarbon from two or more molecules of unsaturated hydrocarbons. So as to inhibit this reaction and establish the conditions for the alkylation reaction, a five- to sixfold excess of isobutane (over that required by the calculations) is used. Thereby the olefins are diluted in the mass of isobutane and react principally with the isobutane.

The temperature of the reaction. The reaction of the

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sulfuric acid alkylation of the BBF with isobutane gives the best results at temperatures between 0 and 10 degrees Centigrade. At lower temperatures there is a noticeable polmerization, the yield of alkylation products diminishes and these products have a wider boiling range. At higher temperatures the reaction of oxidation of the olefins commences thereby fouling the acid and increasing its consumption.

The prescribed temperature conditions for the reaction depends on the type of feed stock and the strength of the acid. Thus, butylene is more actively akylated within the 0-10 degrees Centigrade range; on lowerring the strength of the acid higher temperatures may be allowed.

The alkylation reaction ordinarily proceeds rather rapidly, taking 5 to 6 minutes, but under industrial conditions it is kept going for 20 to 40 minutes to utilize the olefins completely.

The duration of the reaction may be varied according to the feed, the strength of the acid and other conditions.

Strength of the acid, and its consumption. The strength of the sulfuric acid is a very important condition in conducting the alkylation process, the optimum strength being 96 - 97 percent. Stronger acid leads to the reaction of hydrocarbon oxidation and other side reactions, as a result of which the product resinifies and the acid is fouled. More concentrated acid may be used in alkylating propylene than with a butylene feed.

During the alkylation process the strength of the acid is

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lowered by dilution with the water formed by the reaction and with certain other reaction products. Part of the spent acid must therefore be continuously replaced by fresh acid to maintain the necessary acid concentration.

The consumption of acid in effecting the alkylation reaction depends on the type of feed: more sulfuric acid is consumed with light feeds than with heavy ones.

In the alkylation on BF with isobutane the acid consumption amounts to 90-115 kilogram per cubic meter of product.

In practice the ratio of acid to hydrocarbons is held at values such as 1 : 1.

Agitation. To obtain good results in alkylation, the mixture of feed and acid must be thoroughly agitated, otherwise close contact between feed and acid will not be established and this will increase the consumption of acid and reduce the yield of alkylate. In plant operation, therefore, watch must be kept over the normal operation of the circulation and agitation of feed and acid.

Yield and characteristics of the product. Let us see what the approximate conditions are for the alkylation of isobutane with the butane-butylene fractions (Table 43).

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TABLE 43

Reaction temperature, degrees C	0 - 10
Reaction time, minutes	20 - 40
Strength of sulfuric acid, %	98 - 100
Strength of spent sulfuric acid, %	88 - 90
Conumption of acid, kilogram per cubic meter of alkylate	120
Ratio of acid to feed, by volume	1 : 1
Olefin content of feed, % by weight	35 - 45
Isobutylene content of feed, % by weight	15 - 20
Ratio of isobutane to olefin	5 - 6 : 1
Yield of alkylate in % by weight of olefin	150 - 170
Yield of aviation alkylate of end point 150 degrees C, of alkylate	88 - 90

TABLE 44

CHARACTERISTIC OF ALKYLATES AND ALKYLATE AVIATION FUELS

	Alkylate	Alkylate Aviation Fuel
Specific gravity	0.705	0.695
Engler distillation:		
Initial Boiling Point, ° C.	45- 48	---
10%	65 - 85	---
50%	95-105	97
90%	120-140	---
End point	200-210	153
Octane number	91	92
Reid vapor pressure, MM of Mercury	182-208	153
Unsaturated hydrocarbon content	---	Traces

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In working with the propylene fraction, the following approximate alkylation conditions are established:

Ratio of isobutane to propylene	10 : 1
Ratio of acid to feed	1 : 1
Temperature, °C	19 : 20

The reaction of butane with the propane-propylene fraction proceeds more slowly, and the quality of the alkylate produced is lowered: its octane number is 88.

An aviation fuel of low octane number (about 87) is also produced on alkylation of isobutane with the 1-pentene-fraction.

The alkylation reaction may also be conducted with close-cut fractions of gasolines, for example, the alkylation of the light ends of cracked gasoline, boiling between 21 and 9 degrees Centigrade, of the light ends of natural gasoline, etc. The octane number of the aviation fuels produced on using such a feed is lowered to 74-78, but at the same time the feed stock resources are considerably expanded thereby, and the operation of the plant is simplified.

Products obtained by the alkylation of various feed stocks have high antiknock properties, good lead susceptibility and high stability: an alkylate exposed to the sun for a few months does not form gum or change its color.

Table 45 gives data on the alkylation of various olefins.

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TABLE 45

THE ALKYLATION OF OLEFINS BY ISOBUTANE

	C_3 fraction	C_4 fraction	C_5 fraction	Cracked Gasoline (21-71 degrees C)	Cracked Gasoline (21-98 degrees C)
Olefin content, %	30	48	55	--	--
Ratio of isobutane to olefin	8	5	8	8	8
Strength of H_2SO_4, %	98-100	98-100	98-100	98-100	98-100
Ratio by volume, acid to feed	1	1	1	1	1
Temperature, degrees C	29	0-10	10	10	10
Reaction time, minutes	20-40	20-40	20-40	20-40	20-40
Yield of product, % (of olefin) by weight	185	190-200	134	126	115
Percentage by weight of aviation fraction (End Point 149 degrees C) contained in alkylation products	92-95	98-90	90	85-77	--
End Point of alkylate, degrees C	174	171	186	190	195
Octane number of alkylate	88	91.5	87	83	79
Octane number of aviation fraction	89	92	87	82	78

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100. Alkylation with Hydrofluoric Acid

Catalytic alkylation with the addition of hydrofluoric acid is a step forward in the development of the alkylation process, since it has a number of advantages over sulfuric acid alkylation, among which the principal one is the expansion of the raw material base for the production of blending components for aviation fuel, in view of the fact that propylenes, butylenes and amylenes (separately or together) all react with the same ease in the presence of hydrofluoric acid.

A second advantage of this method is the possibility of conducting the alkylation reaction at room temperature, while sulfuric acid alkylation requires the reaction products to be cooled to 0-10 degrees Centigrade. However, in this method only an insignificant amount of cooling is needed to carry off the heat liberated by the reaction.

A third advantage is the possibility of completely regenerating the catalyst and reusing it.

The principal equipment of a hydrogen fluoride alkylation plant consists of a reaction chamber, an acid settling tank, an acid regeneration column, a rectifying column for separating the gases into propane, butane and isobutane, and a rerun column.

The flow sheet (Figure 128). The charge stock (the olefin fraction) is diluted to the required norm with isobutane and passed through the column 2, filled with bauxite, for dehydration to remove any traces of moisture. The dehydration of the feed is necessary, since even traces of moisture impair the quality of anhydrous HF.

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The circulating isobutane 4 is added to the dehydrated feed and the mixture passed into the reaction chamber-mixer 3. Anhydrous hydrofluoric acid, first mixed with the circulating isobutane, is also introduced into the reaction chamber mixer. The dilution of the acid is necessary to prevent polymerization, which readily takes place in olefins in the presence of HF. HF boils at 19.4 degrees Centigrade, and under the reaction conditions, i.e., at 20-40 degrees Centigrade, it is present in the vapor state and therefore mixes readily and intimately with the hydrocarbons. The structure of the reactor is calculated to assure the rapid and vigorous mixing of the reacting substances. The pressure within it is held between 7 and 10 atmospheres. This means that the reacting substances are present in the liquid state, thus facilitating their intimate contact.

The mixture of reaction products and acid flows from the reactor to the acid settler 6. The waste acid 5, containing a certain amount of hydrocarbons, is discharged from the bottom of the settler.

The spent acid is restored in the acid recovery plant 8. For this it is first distilled off, freed from all admixtures, and again recycled into the system (7) for reuse. The reaction products (alkyls) taken overhead from the acid settler 6, contain up to 1 percent HF and a certain quantity of organic fluorine compounds. The alkylate is freed from the admixtures in the regenerating plant 10, consisting of a column for evaporating off the HF and packed columns filled with bauxite.

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The mixture is passed through the HF column (not shown in the diagram) which operates under 14 atmospheres pressure. The light hydrocarbon and acid vapors are driven off at the top of this column; the acid is easily separated from the hydrocarbons after the mixture has been condensed and cooled below 19.4 degrees Centigrade. Alkylate still containing some fluorides are drawn off at the bottom. To remove these fluorides the alkylate is passed through bauxite columns, from which it emerges freed of admixtures. The bauxite columns operate alternately, with each one taken off stream when the fluorides amount to 20 percent by weight of the bauxite. The fluorides are then decomposed into hydrocarbons and HF. The recovered acid is returned to the system at 9. In practice all the HF is recovered, and therefore the addition of fresh acid is almost unnecessary.

From the acid-recovery unit the alkylate passes to the isobutane column 15, yielding isobutane and propane overhead and alkylate from the bottom. The mixture of isobutane and propane is separated at the propane column 14 into the propane 13 and the isobutane.4. The isobutane is recycled into the system, while the alkylate passes through two more columns: the butane column 16 to drive off the butane, and the rerun still 19, from which, after rectification, the aviation alkylate 18 is taken overhead and the heavy alkylate 20 at the bottom.

Reaction conditions. Feed. The olefin feed may consist of propylene, butylene, or 1-pentene, all of which readily enter the alkylation reaction singly or in mixture. Isobutane is the iso-paraffin feed that is mostly used.

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The ratio of isobutane to olefins. To inhibit the polymerization reaction, a five- to eightfold amount of isobutane is generally used.

The temperature conditions of the reaction. The alkylation is usually conducted at 24-46 degrees Centigrade, depending on the temperature of the cooling water. The process therefore takes place at higher temperatures in summer than in winter.

Pressure. Alkylation takes place in the reactor under pressures of 7-10 atmospheres, i.e., in the liquid phase, which is necessary to assure good contact between the reactants.

Strength and consumption of the acid. The strength of the acid exerts a very great influence on the alkylation process. For instance, in alkylating isobutane and BBF in the presence of 100 percent acid, 50-60 percent of the reaction products consist of octanes, half of which is isooctane (with octane number 100). The HF content of the alkylate in this case is only 0.1 percent. But in alkylating the same feed in the presence of 90 percent acid, octanes comprise 48 percent of the hydrocarbon layer, while the HF content rises to 1 percent. In the presence of 75 percent acid, alkylation does not take place at all.

Thus, it is best of all to use 100 percent, or anhydrous, HF.

The acid is fully recovered without losses, and is then recycled into the system for further use. No more than 0.2 percent HF need be added, so that, in spite of the high cost of HF, the process is still very economical.

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Troubles in the operation of a unit. Spar acid (a solution of HF) destroys very many materials. However, anhydrous HF, or concentrated acid (over 80 percent), does not act on steel, copper or Monel metal. In working with HF, however, materials containing silicon in any form cannot in any case be used (for example, glass, cement, silicious pig iron), since the acid will easily destroy them. Neither can wood, rubber and most plastics be used. The equipment coming in contact with HF is mostly manufactured of carbon steel; safety valves and control instruments are covered with rustless steel. Equipment in contact with dilute HF is manufactured of Monel metal. Pumps are made with steel shells and wheels. Asbestos gaskets, covered with copper braiding, are used. Forged steel fittings are used on all piping, with packing exclusively of iron or copper. The thread of screw couplings must be clean, and any welding must be high grade. HF boils at 19.4 degrees Centigrade, so that it must be stored in insulated tanks to prevent evaporation.

Questions involving safety are very important in work with HF. The personnel of the unit must be well acquainted with the properties of HF and must know how to act quickly and decisively in case of danger.

101. Thermal Alkylation

Thermal alkylation is conducted at high temperatures (500-510 degrees Centigrade) and high pressures (from 200 to 350 atmospheres). High pressure is necessary to prevent the hydrocarbons from being cracked. A high isobutane concentration is maintained in the feed to prevent polymerization of the olefins.

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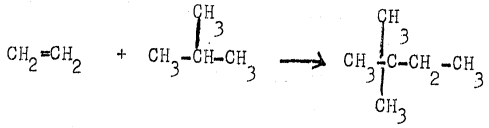
The results of thermal alkylation resemble those of sulfuric acid alkylation, but differ in the following respects:

In sulfuric acid alkylation, isobutylene reacts readily, butylene less readily and ethylene very slightly; in thermal alkylation, on the contrary, ethylene reacts most readily of all.

In sulfuric acid alkylation only the isoparaffin hydrocarbons become alkylated, while in thermal alkylation the normal hydrocarbons also react readily.

These features of thermal alkylation expand the field of the raw material resources for the production of high-octane blending components. The alkylate yield from thermal alkylation is high, and only a little gum and gas is formed (regardless of the properties of the feed used).

In spite of the list of favorable features, thermal is not widely used because of the extreme process conditions and the complicated and heavy equipment required. A full-scale industrial thermal alkylation plant provides for the production of a high-octane hydrocarbon -- neohexane -- from ethylene and isobutane:



Ethylene + Isobutane = Neohexane

The manufacture of neohexane takes place in two stages. In the first one, the ethane-propane fraction is subjected to pyrolysis in a pipe still at 760 degrees Centigrade and normal pressure to

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produce ethylene. The pyrolysis gas, rich in ethylene, then enters an absorber operating under 56 atmospheres pressure and an isobutane reflux. The isobutane-ethylene mixture is delivered by a compressor into an alkylation pipe still operating at 510 degrees Centigrade and 315 atmospheres pressure. An excess of isobutane is pumped into the ethylene-isobutane fraction, before it enters the pipe still, to prevent polymerization. The reaction products are then passed through a depropanizer, a debutanizer, and a rerun column. A product containing up to 50 percent neohexane is thus produced. Pure neohexane is then separated by a rectifying column with sharp rectification.

Neohexane boils at 49.7 degrees Centigrade, and in the pure state has an octane number of 95. It has very good lead susceptibility: with 3 cc of ethyl fluid it behaves exactly like isooctane (with octane number 100), also on addition of 3 cc of ethyl fluid. Its lead susceptibility is therefore higher than that of isooctane.

102. The Catalytic Alkylation of Aromatic Hydrocarbons

Copper pyrophosphate on kieselguhr and phosphoric acid on kieselguhr are widely used today as catalysts in alkylation plants.

As already stated, during recent years a large amount of the high-octane aromatic hydrocarbon isopropylbenzene, more commonly known as cumin, has been produced. Cumin is employed only as a blending component for aviation fuel. Its specific gravity is 0.863, boiling point 146-175 degrees Centigrade, and octane number 99.5. Blended with aviation fuel it behaves like a hydrocarbon of

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octane number 124.

A flow sheet for cumin production, using phosphoric acid on kieselguhr as a catalyst, is given in Figure 129.

The Flow sheet. The propane-propylene fraction is mixed with benzol, heated first in the exchanger 1, then in the furnace 2, and passes, at 260 degrees Centigrade and 50 atmospheres pressure, into the catalyst-filled reactor 3. The temperature of the mixture is increased by the reaction, and benzol is therefore circulated through the reactor at various levels to hold this temperature constant.

The reaction products are taken from the bottom of the reactor, pumped through the feed-heat-exchanger, and then passed to the depropanizer 4 to remove the propane and the unreacted propylene. The mixture of alkylate and benzol passes from the bottom of the depropanizer into the benzol column 5 to drive off the unreacted benzol, which is then mixed with fresh benzol and delivered, as already described, for mixture with the propane-propylene fraction, and then to the reactor.

The alkylate is routed from the bottom of the benzol column to the rerun column 6 for separation into light and heavy alkylates.

The following are the approximate operating conditions in the alkylation of benzol with propylene:

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Temperature, ^{0} C	260
Pressure, atmospheres	50
Mol ratio of benzol to propylene	10 : 1
Rate of reaction	3 volumes of liquid (per hour) per volume of catalyst
Degree of propylene, %	85
Yield of cumin in % of propylene reacted	200 - 205
Consumption of catalyst in % of cumin	0.3

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CHAPTER ELEVEN

THE PRODUCTION OF OTHER PETROLEUM PRODUCTS

103. Petroleum Asphalts

The demand for asphalts is very great. A few hundred thousand tons is required merely for the annual spreading on the roads. This demand cannot be met by the extration of natural asphalt alone. The petroleum industry therefore produces artificial asphalts, or, as they are termed, petroleum asphalts.

Petroleum asphalts are produced by two methods:

(1) On distilling off the lubricating oil distillates from oil- tar in vacuum stills, an asphalt termed residual asphalt may be obtained in the residue;

(2) On blowing air through high-tar residues (tars or cracked residues) heated to 250-300 degrees Centigrade in vertical or horizontal stills, a so-called oxygenated asphalt is obtained.

The essential feature of the second method consists in the oxygenation of oils and the conversion of tars to asphaltenes. In one of the batch process plants, 88 percent of mark III asphalt is obtained by oxygenation of tar (at 270-300 degrees) with air. A single run (or cycle) of a still takes 50-60 hours. The fuel consumption amounts to 6 percent of the amount of tar.

There are five marks of petroleum asphalt which differ mainly in the following properties:

(a) Ductility or extensibility, which is 100 centimeter for mark I asphalt, 50 centimeter for mark II, 40 centimeter for

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mark III, 3 centimeter for mark III, and 1 centimeter for mark V.

(b) Penetration, which is 160-200 for mark I, and then respectively 90-120, 40-70, 20-40, and not above 10 for the last or mark V.

(c) Softening point (by the ring-and-ball method), which is 30 degrees Centigrade for mark I, and then respectively 40 degrees Centigrade, 50 degrees Centigrade, 70 degrees Centigrade and 90-100 degrees Centigrade for the remaining four marks.

The first three marks of asphalt may be obtained as residue from the distillation of mazut in vacuum plants, while marks IV and V are obtained exclusively by the oxygenation of heavy residues in stills.

Marks I, II and III are mostly used to cover roads and as insulating material. Mark IV is used in the roofing industry for roofing cement and waterproofing. Mark V is used to manufacture rubberoid (asphalt-covered cardboard), plastic cement, cable cement for electric insulation, etc.

Asphalts of the first three marks are transported in special asphalt tank cars, well insulated on the outside and provided with steam coils to heat the bitumen on discharging it. Marks IV and V are shipped in kegs, special kraft-paper sacks, and also in bars, blocks and slabs. Asphalt should be stored on asphalted platforms under sheds.

Besides these five marks of asphalt, special asphalts are also manufactured with softening points 110-125 degrees, 125-135 degrees, and 150 degrees Centigrade (rubrax). They are

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used in the colored varnish and rubber industries.

104. Paraffin

The paraffins are composed of large plate- or band-shaped crystals. Their boiling range is from 370 to 560 degrees Centigrade, and they can therefore be distilled along with the lubricating oil distillates.

The Production of Paraffin consists of the following operations: destillation of mazut with separation of a wax stock, crystallization of this distillate, filtration, sweating the paraffin mass and refining the raw paraffin.

Paraffin mazut is distilled in shell-still or pipe-still units. Even a low percentage of ceresin impairs the quality of paraffin and the distillation is therefore so conducted that no ceresins pass over in the distillate with the paraffin.

The wax stock separated from Grozniy paraffin crude and making up 30-32 percent of that crude, is characterized by the following properties: specific gravity, 0.860 - 0.870; viscosity, E_{50} = 1.7 - 1.9; solidification temperature, between 25 and 30 degrees Centigrade; it contains up to 20 percent paraffin with an average melting point of 50 degrees Centigrade.

The wax stock is cooled to a temperature of +2 to +6 degrees Centigrade and pumped through a filter press. After pressing, a paraffinic press cake termed cheese paraffin is obtained. Cheese paraffin contains 20-50 percent oil. To remove the oil, the cheese paraffin is subjected to the process of sweating, which consists of slowly heating the cheese paraffin,

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which has been cooled to solidification, in a sweating chamber (Figure 130). Droplets of oil are thereby exuded by the paraffin and flow off through a sieve in the bottom of the chamber. The sweating temperature is so adjusted that the paraffin reaches the required melting point by the end of the sweating.

Paraffin produced in the sweating chamber may be used in the manufacture of candles. Paraffin after treatment with sulfuric acid is so used, as well as in the manufacture of perfumery, vaseline and creams, for impregnating paper, in the chemical industry, etc.

105. Petroleum Coke

Petroleum coke is the product obtained by coking liquid residues, tar, cracked residue, pyrolysis residue. It contains 90-93 percent hydrocarbon, hydrogen, and 1-1.5 percent sulfur, nitrogen and oxygen. It is ash-free and is therefore an excellent material for the manufacture of the electrodes used in the aluminum industry.

The best stock for producing coke is the residue from the distillation of heavy crudes, or from the pyrolysis furnace, which yields up to 20-25 percent coke. A 3-10 percent yield is obtained from the residues of paraffinic crudes at 500 degrees C.

The process of producing coke in iron batch-stills consist of the following steps. The stock is charged into a horizontal iron still 60-75 cubic meters in capacity, and is distilled to coke without the use of steam. To save time the stock is first preheated to 350-400 degrees Centigrade before charging. At 360-400 degrees Centigrade, the stock begins to decompose; the decomposition

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products and the light ends of the stock are driven out of the still. The still temperature reaches 760-780 degrees Centigrade by the end of coking. After the coking process has been completed, open steam is passed into the still to cool the coke and remove the decomposition products.

When the temperature in the still has been lowered to 175-200 degrees Centigrade, the upper hatch is opened, and at 60-80 degrees Centigrade the coke is discharged. The coking cycle takes 50 hours.

The stills operate under very severe conditions, and their bottoms must therefore be frequently replaced.

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CHAPTER TWELVE

THE TREATMENT OF DISTILLATES AND THE PRODUCTION OF COMMERCIAL OIL PRODUCTS

In almost all cases, the distillates produced on the distillation of crude or mazut, or by cracking or pyrolysis, are not finished products since they contain various harmful substances which act adversely on metals, producing carbon deposits, forming coke, etc.

Such substances may consist of:

resinous and asphaltic substances; when petroleum products containing these substances are used, carbon and coke are deposited; petroleum acids which act corrosively on metals; on combustion, carbon is deposited; nitrogen compounds; mineral admixtures and ash; unsaturated hydrocarbons.

After these harmful substances are removed from the distillates, the color of the petroleum products is improved, their content of coke, ash and gum is reduced, and they become more stable in storage. The harmful substances are removed from distillates by treatment, or refining. Ordinarily the following treatment methods are used: sulfuric acid; alkali - caustic soda or calcined sodium carbonate; adsorption -- by clays able to absorb (adsorb) harmful substances; selective -- by solvents in which the solubility of the farmful substances is high and that of the petroleum product itself is low.

Other methods of treatment less frequently employed are: desulfurizing light petroleum products by sodium plumbite, or hypochlorite; treatment with salts -- zinc chloride, aluminum chloride; treatment by menas of hydrogenation.

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I. THE ACID-ALKALI TREATMENT OF DISTILLATES

106. The Action of Sulfuric Acid in Treatment

The action on distillates of sulfuric acid is the oldest method and at the same time the one most widely used. It may be applied to all distillates requiring treatment without exception. Sulfuric acid, which acts chemically on almost all harmful substances only slightly affects the petroleum product itself.

Treatment of petroleum products with sulfuric acid is often conducted at units provided with mixing tanks (Figure 131).

Sulfuric acid is a heavy liquid with a specific gravity up to 1.84. Dilute sulfuric acid (of strength up to 75 percent vigoruously attacks cast iron and pig iron, but does not attack lead. Sulfuric acid of 80 percent and over, does not attack cast iron or pig iron but attacks lead. Taking advantage of this property of sulfuric acid, the treatment of distillates is conducted in crude cast iron equipment for treatment with concentrated acid, while the washing and the alkali treatment, when the acid becomes dilute, is conducted in lead-lined apparatus. Let us consider the interaction of concentrated sulfuric acid with hydrocarbons and with various harmful substances.

Paraffin and naphthenic hydrocarbons do not react with sulfuric acid under ordinary conditions of treatment, but on treatment with fuming sulfuric acid the latter react to form sulphonaphtenic acids.

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The aromatic hydrocarbons form sulfonic acids fairly easily with sulfuric acid, but under the conditions of sulfuric acid treatment, when the sulfuric acid in the first place reacts with other and less stable substances, the aromatic carbons are almost unaffected.

The unsaturated hydrocarbons react actively with sulfuric acid, forming polymers, acid and neutral esters.

Under sulfuric-acid treatment, the diolefins are the first of the hydrocarbons to enter into the polymerization reaction. This property is very important in the treatment of cracked gasoline, since it is desired to remove these diolefins, and to preserve the diolefin hydrocarbons in the product.

The petroleum acids are readily soluble in sulfuric acid, and also react chemically with it. However, when there is a considerable content of these acids in the distillate, it is better to conduct a preliminary leaching of the distillate to remove these acids and to use them for the preparation of naphthenic soap.

The sulfur compounds dissolve in sulfuric acid with partial oxidation. If the content of hydrogen sulfide is high, it is expedient to remove it by a preliminary leaching. Resinous and asphaltic substances readily react with sulfuric acid to form heavy products consisting of asphaltenes, sulfonic acids, etc, which settle to the bottom of the tank in the form of acid sludge. Resinous and asphaltic substances are also readily soluble in sulfuric acid. As a result of the sulfuric acid treatment of distillates, three products are formed: sulfur dioxide,

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which escapes into the atmosphere; acid sludge, which settles on the bottom of the apparatus, and the refined product.

The influence of various conditions of the treatment process -- the feed stock. According to whether a straight-run gasoline, a cracked gasoline or high-viscosity tarry lubricant oil distillates are being treated, all the other conditions are selected so as to be optimum for the given chemical composition of the product.

$H_2 SO_4$ with a strength of 60 percent readily reacts with unsaturated hydrocarbon and resins; to remove other substances, a stronger acid (92-96 percent) is necessary, which is termed oil of vitriol in refinery practice.

98 percent acid called monohydrate, or fuming sulfuric acid (sulfuric acid containing free sulfuric anhydride, SO_3) called oleum, is used in some cases for special treatment (turbine, transformer and white oils).

The quantity of acid used for treatment depends on the chemical composition of the product to be treated; 1-3 percent is enough for treating the light petroleum products, 5-10% is required for treating the lubricant-oil distillates, while in some cases as much as 50-60 percent of sulfuric acid may be necessary.

At higher treatment temperatures, sulfuric acid easily reacts with all substances, but to avoid affecting the aromatic and naphthenic hydrocarbons, the lowest possible treatment temperature must be maintained. Its reaction with resins and

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unsaturated hydrocarbons is thereby unaffected.

In treating certain products (for instance, illuminant kerosene or galosh-benzine, in which aromatic hydrocarbons are harmful) a higher temperature is maintained.

Lubricating oils have high viscosity at low temperatures. In this case to facilitate mixing the temperatures must be held between 40 and 60 degrees Centigrade.

The better the sulfuric acid is mixed with the distillate, the better will the treatment process proceed. Ordinarily the mechanical or air-jet method of agitation is employed. Air agitation is used in the treatment of lubricating oils, but it is entirely inapplicable in the treatment of the light petroleum products, since it leads to product losses. A continueous process of refining with mechanical agitation is most often used with light petroleum products.

107. The Action of Alkali in Treatment

Caustic soda, NaOH, and soda ash, Na_2CO_3, is usually employed for the treatment of distillates.

Dry caustic soda is a white crystalline mass of specific gravity 2-2.1. It avidly absorbs moisture from the air, for which reason it is stored in hermetically closed iron drums. These drums are opened at treatment plants and the caustic soda is dissolved in water to the required concentration. Soda ash is a white powder and is transported in casks or bags.

Alkali treatment is sometimes employed independently to

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remove sulfur compounds or petroleum acids, but as a rule it is used to supplement sulfuric acid treatment. The object of alkali treatment is to neutralize the acid compounds contained in the distillate or formed during sulfuric acid treatment. The former comprise the petroleum acids (already mentioned) and sulfur compounds (hydrogen sulfide, mercaptan, etc), and the latter comprise are the sulfonic acids and certain other sulfur compounds. Traces of sulfuric acid also remain in the distillates and must be neutralized. As a result of the action of alkali on acid compounds, the sodium salts of these compounds are formed. These salts are readily soluble in water and therefore settle to the bottom of the mixer, thus forming alkali liquor. Emulsions may be formed during the acid treatment of distillates, especially of lubricant-oil distillates. They are formed with particular ease on treatment with strong alkalis. For this reason alkali of 3-6° Beaume is used in this case. The light petroleum products have less of a tendency to form emulsions, and therefore stronger alkali solutions are used for them (7-10° Beaume, and even as strong as 15° Beaume).

Since caustic soda is expensive and in short supply, refineries try to replace it by other alkalis -- soda ash, lime, ammonia. On neutralization with lime, calcium salts are formed which are not readily soluable in water, and therefore part of them remain in the finished product, increasing the ash-content of the oils.

Soda ash is less active than caustic soda and is therefore used only in treating gasoline and kerosene distillates. It is better to use a mixture of equal parts of soda ash and caustic soda.

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In working with chemicals (acids, alkalis) it is necessary to observe the established safety regulations if accidents are to be avoided.

Sulfuric acid coming in contact with the skin causes burns. Water should not be used to wash acid from the skin; it should be wiped off with a handkerchief, towel, absorbent cotton, etc, and only after this has been done should the burn be washed off with a weak solution of sodium carbonate, after which it should be anointed with vaseline and bandaged.

When acid is mixed with water, the acid must be poured into the water; under no circumstances may the water be poured into the acid (to avoid burns from spattering of acid). The mixing of water and acid should be done in a closed apparatus.

In working with acids, gauntlets and overalls must be worn. It is forbidden to make any repairs on apparatus in which there is acid.

Strong alkalis also burn the skin, and therefore the established rules must be carefully observed in handling them.

108. The Treatment of Gasoline and Ligroin Distillates

The principle harmful substances that must be removed from straight-run gasoline distillates are the sulfur compounds. Gasolines produced by distillation on the sulfur-bearing crudes of the Second Baku have a particularly high content of sulfur compounds.

Below is the approximate sulfur-compound content of gasoline

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distillate produced from sulfur-bearing crude (calculated in percentages of the crude):

Hydrogen sulfide	0.2%
Mercaptan	0.2%
Sulfides, disulfides and othersulfur compounds	0.3 to 0.5%
Total sulfur	0.7 to 0.9%

The sulfur compounds are divided into two groups: (1) active (hydrogen sulfide, mercaptan, free sulfur) and (2) inactive (sulfides, disulfides, thiophene).

Hydrogen sulfide corrodes a tank very actively. It is a permanent and poisonous gas. The reaction between hydrogen sulfide and alkalis takes the following form:

$$H_2 S + 2 NaOH = Na_2 S + 2 H_2O.$$

The mercaptans are also very injurious and must be removed from gasoline:

$$CH_3 SH + NaOH = CH_3SNa + H_2O.$$

The other sulfur compounds we have mentioned are neutral and their presence in gasoline, in amounts not exceeding 0.1-0.2 percent, is allowable.

The presence of active sulfur compounds in gasoline is determined by the so-called doctor test and the copper-strip test.

The doctor test is a simple operation that can be performed by the refiner at the treatment plant. 10 cubic centimeter of the gasoline, 5 cubic centimeter of lead plumbite and 5 percent of

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sodium plumbite (doctor solution) are poured into a graduated 50 cubic centimeter cylinder and vigorously shaken for 15 seconds. A spoonful of flowers of sulfur is then sifted into the cylinder and it is again shaken for 15 seconds. On standing, the sulfur will form a layer between the heavy doctor solution and the lighter gasoline.

Ordinarily, flowers of sulfur is yellow. If this color remains unchanged, the test is considered negative, meaning that the gasoline does not contain active sulfur compounds. If the color changes from yellow to orange or brown, the test is considered positive, meaning that the gasoline does contain active sulfur compounds.

The doctor test indicates the presence of hydrogen sulfide and the mercaptans, but does not reveal the presence of free sulfur, which is also an actively corrosive substance, and therefore the copper-strip test must also be performed. For this purpose a strip of red copper (of the prescribed size) is kept in the gasoline for 3 hours at a temperature of 50 degrees Centigrade. If the surface of the strip darkens, this indicates the formation of a layer of copper sulfide on it.

The continuous treatment of gasoline and ligroin (Figure 132). A gasoline treatment plant consists of 6 horizontal setting tanks and 6 injector-mixers (before each settling tank). The mixer is a section of 8-inch pipe, containing 52 half-inch tubes, welded at one end to a tube sheet in the large pipe (the other ends of the tubes remaining open). Two 3-inch supply-pipes are welded to the large pipe and the gasoline is fed through them.

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The settling tanks 2, 3 and 4 serve to remove the acid from the product being treated while the settling tanks 5 and 6 serve to remove the alkali liquor and settling tank 7 is used for the final settling of the refined product.

The settling tanks 2 and 3 are charged with sulfuric acid of 92-96 percent strength while tanks 5 and 6 are charged with alkali solution 10 degrees Beaume).

The gasoline is delivered by a pump into the injector 13, where it is mixed with the sulfuric acid drawn from the settling tank 2. The mixture of gasoline and acid then goes to tank 2, where the acid is settled off and recycled to the injector. In this way the acid is in continuous circulation. The acid gradually becomes spent, i.e., its strength is reduced. After 10-12 hours it is replaced by acid from settling tank 3, in which the acid is more slowly exhausted, since this acid is reacting with a partially refined product. The product passes from tank 2 into the injector tank 14 for mixing with stronger acid (from tank 3) and then into tank 3 itself. In tank 3, the acid is replaced by fresh acid (after 10-12 hours have elapsed). From tank 3 the acid product passes to tank 4 for final settling of the residues of acid and acid sludge. In the same manner, the gasoline passes through injectors 10 and 11 and settling tanks 5 and 6 for alkali treatment.

The spent alkali is replaced after every 8-10 hours of operation. The gasoline is finally freed from the alkali residue in the last settling tank 7.

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The consumption of acid at the plant amounts to 1 percent, and that of alkali to 0.03-0.04 percent, in terms of dry alkali.

The quality of the work of a plant is determined by its productivity, the properties of the refined product, the consumption of reagents and the condition of the equipment.

It is especially important that the injector (Figure 133) should operate normally. If the injector operates badly, the quality of the refined gasoline will be adversely affected and the reagent consumption will be increased. A sample of the product is taken from the cock of the injector several times during a shift. If operation is normal, the sample will separate in the test tube into two layers, and the quantity of the reagent should not be less than 30 percent by volume of the gasoline. When the injector is working badly, however, none of the reagent at all will appear in the sample, or only a very insignificant amount of it. In this case, the reserve mixer is put on stream.

In treating a gasoline distillate produced from sulfur-bearing crudes, the consumption of sulfuric acid reaches 4 percent and more. In the sulfuric acid treatment of such a distillate, a considerable amount of compounds boiling at a temperature of over 200-225 degrees Centigrade are formed. For this reason the gasoline obtained must be redistilled at a rerun plant, which reduces sulfur content from 0.5-0.6 percent to 0.08 percent.

109. Treatment of Pressure Distillate

The principle harmful substances that must be removed from pressure distillate (cracked distillate) are the sulfur compounds,

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the resins and the diolefins. A number of methods for treating pressure distillate are known:

(1) Acid-alkali treatment followed by redistillation,

(2) alkali treatment with the addition of anti-oxidents (inhibitors),

(3) vapor-phase treatment with clays followed by rectification of the treated gasoline,

(4) hypochlorite and plumbite treatment.

Acid-Alkali Treatment

Sulfuric acid with a strength not over 80 percent is used for treating pressure distillate. The process of treatment is conducted continuously in vertical columns. One of these plants is equipped with 5 columns (Figure 134): an acid column, two settling columns, a wash column and an alkali column. The reagents are mixed with the pressure distillate in the injectors.

The pressure distillate is delivered by a pump first to the injector and then to the bottom of the acid column. To prolong the reaction time, the mixture is passed through an acid layer in the column. The level of the acid is held at about 2 meters. As the pressure distillate moves upwards, some of the sulfuric acid and acid sludge are simultaneously settled out.

From the top of the acid column, the acidified pressure distillate first enters the first settling column and then the second one, where the acid and acid sludge is removed by settling. The water in the wash column is kept at the 2-meter level. The acid product, traversing the layer of water, is finally freed from acid and acid sludge.

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After washing, the product is subjected to alkali treatment in the alkali column, where the last traces of acid and acid compounds are removed.

The consumption of acid for treatment of pressure distillate produced from nonsulfur-bearing crude, amounts to 0.8-1.2 percent of the quantity of pressure distillate.

Sulfuric Acid Centrifugal Treatment.

In the treatment of distillates obtained from sulfur-bearing crudes, the consumption of sulfuric acid reaches 6 percent of the amount of the distillate. When such an amount of sulfuric acid is used in treating cracked and reformed distillates, the olefin hydrocarbons actively enter the reaction, which leads to large distillate losses.

In order to eliminate only harmful substances from the distillates -- sulfur compounds, resins and diolefins -- a method is used in which the contact between the acid and the distillate occurs instantaneously and the reaction takes place very energetically. This is effected in special dispersion contractors; the separation by settlement also takes place very rapidly in centrifuges, in a few seconds.

The principle equipment of the three-stage sulfuric acid centrifugal plant consists of rapid-pass contactors (mixers) centrifuges, separators and coolers.

The mixer of the plant (Figure 135) consists of a cylindrical chamber, inside of which a screw propeller, a group of

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baffles and a circulation tube are mounted. The distillate and acid are delivered under pressure to the mixer. The mixture is circulated and intensively agitated inside the mixer.

The centrifuge of the plant (Figure 136) operates at rotor speeds of about 7500 revolutions per minute. The complete separation of the acid sludge from the pressure distillate is thereby achieved.

The flow sheet for this process is shown in Figure 137. The raw distillate, after first being treated with alkali to remove the hydrogen sulfide, and heated to 27-28 degrees Centigrade in the exchanger 4, enters the first mixer, where it is treated with sludge from the second stage of the treatment.

The contacting (mixing of the reagent with the distillate being treated) in the mixer takes only a few seconds, after which the mixture enters the centrifuge of the first stage, where the acid sludge is separated and removed from the plant. This operation also takes only a few seconds.

The distillate is then delivered to the second stage of treatment, where it is treated in the second mixer 3 with acid sludge from the third stage of the treatment, and then enters the centrifuge.

As has already been stated, acid sludge of the second stage is used for treatment of the distillate in the mixer of the first stage. In the mixer of the third stage the distillate is treated with fresh sulfuric acid, while the acid sludge of this stage is utilized in the mixer of the second stage.

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The treated distillate after the third stage is neutralized by alkali and washed with water in the ordinary continuous-operation units.

Below are data on the operating conditions of the plant.

TABLE 46

Name of Product	Consumption of acid, %	Strength of acid, %	Temperature of distillate, Stages			Temperature of acid sludge, Stages			Pressure, atmospheres, Stages			Mixing time, seconds, Stages			Acid sludge, %
			I	II	III	I	II	III	I	II	III	I	II	III	
Straight-run gasoline	5.2	91.7	27	31	26	45	50	54	0.4	0.3	0.3	2.8	5.6	8.4	7.4
Reformed gasoline	2.4	86.5	28	30	26	56	53	57	0.5	0.3	0.3	3.2	6.4	9.6	3.61

As a result of the treatment the sulfur content of the distillate is reduced from 0.4 percent to 0.1 percent.

Cold Sulfuric-Acid Treatment

At reduced temperatures the reaction between sulfuric acid and the substance being treated is slowed, while their solubility in sulfuric acid is increased. In recent years this property of sulfuric acid has been utilized for the treatment of gasolines. The flow sheet of the treatment in this case remains substantially unchanged and only exchangers and coolers are added.

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The treatment takes place at a temperature as low as 7 degrees Centigrade. This method makes it possible to save 30-50 percent of the sulfuric acid and reduces distillate losses considerably (Table 47).

TABLE 47

Indices	Cold treatment	Hot treatment
Sulfur content in distillate boiling up to 229° C	0.345	0.345
Consumption of sulfuric acid, %	2.76	3.7
Losses in treatment, %	2.6	2.9
Losses from polymerization, %	2.8	7
Yield of gasoline with end point 207° C, %	94.6	90.2
Sulfur content of gasoline	0.106	0.106

The Redistillation of Treated Pressure Distillate

As a result of treatment with sulfuric acid, the unsaturated hydrocarbons and sulfur compounds in pressure distillate are converted into high-boiling substances, which raise the end point of the treated product 20-30 degrees Centigrade, impart a yellowish-brown color to it and raise its specific gravity. Thus, the end point of a certain pressure distillate before treatment was 241 degrees Centigrade, but after treatment it was increased to 268 degrees Centigrade, while the end point of a merchantable cracked gasoline must not be over 225 degrees Centigrade. For this reason, to obtain a merchantable gasoline, the treated pressure distillate must be redistilled. This is done in shell still or in pipe stills.

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The throughput of rerun plants is ordinarily small. These plants are designed only to distill the pressure distillate produced at the refinery in question.

The principal equipment of such a plant includes a pipe still, a rectifying column, heat exchanger, mixture condensers, coolers and a dehydrator. Let us study the flow sheet of such a plant (Figure 138). The treated pressure distillate is heated in the pipe still to 180-200 degrees Centigrade and then enters the column. Steam is introduced at the bottom of the column. After traversing the mixture condensers, the cooler and the dehydrator, the finished cracked gasoline goes to storage. Polymers (15-20 percent of the pressure distillate) are taken off at the bottom of the column.

Cracking plants that process sulfur-bearing crude, produce, as we hafe seen, cracked distillates with high contents of sulfur compounds. In order to prevent the decomposition of these compounds during redistillation, and to establish the smoothest possible operating conditions, two-flash plants with the charge heated by steam (Figure 138) are used in modern refineries. The plant consists of an atmospheric column where the light ends of the gasoline are taken off, a vacuum column where the heavy ends of the gasoline are taken off, stripping stills, exchanges, condensers, mixers and other equipment.

The treated cracking distillate 1 is heated in the exchanger 2 by the heat of the vapors leaving the atmospheric column 4, and in the steam-stripping still 3 by the superheated steam 17, and then enters the column 4. The light fractions of the gasoline are

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routed through the heat exchanger 2 into the condenser-cooler 7, and then to the receiver 8, whence they are pumped into the commercial storage tank 16.

The residue from the column enters the steam-stripping still 5, where a certain amount of the light fractions are stipped, and then passes into the tank 6 from which it is pumped into the vacuum column 9. The heavy fractions of the gasolines are taken off overhead from the vacuum column and pass through the condenser 11 and the receiver-dehydrater 12 into the storage tank 14. Before the residue from the column is removed from the plant, it is passed through the stripping still 10 for supplementary stripping of the gasoline fractions carried along.

A disadvantage of this method of treating pressure distillate with sulfuric acid followed by redistillation is that (1) the total losses during treatment and redistillation amount to as much as 10-15 percent of the pressure distillate; (2) as a result of the polymerization of the diolefins, and of part of the olefins, the antiknock properties of the gasoline are impaired.

The Production of Inhibitor Gasoline

There is a method of treating pressure distillate in which the losses are considerably smaller than with the sulfuric acid treatment, while the antiknock properties of the product are not impaired. This is the alkali treatment of pressure distillate, followed by the addition of an anti-oxidant, termed an inhibitor (which means slowing agent). The Alkali treatment frees the pressure distillate from hydrogen sulfide and the mercaptans.

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The diolefins may be left in the gasoline if their tendency to oxidation and polymerization is retarded, which is precisely what is accomplished by adding the inhibitor. As an inhibitor, USSR refineries use the fraction of wood tar (from beech trees) with a boiling range of 240-300 degrees Centigrade. The consumption of tar is about 0.06 percent of the gasoline. The process of producing an inhibitor gasoline in two-coil cracking plants consists of the following steps. An alkali solution is pumped into the pressure distillate before it enters the stabilization column. After the alkali treatment the gasoline is subjected to stabilization. The stabilized gasoline is then mixed in mixing tanks with a solution of the inhibitor in gasoline. Gasoline containing this inhibitor may be kept in storage tanks for 6 months.

110. The Treatment of Kerosene Distillate

The harmful substances in kerosene distillate are petroleum acids and resins. Kerosene produced from nonsulfur-bearing crudes does not usually contain active sulfur compounds.

The acid-alkali treatment of kerosene distillate is conducted in batch mixers or in a continuous treating plant.

The mixers (Figure 139) consists of iron tanks with conical bottoms, each holding 100-300 cubic meters.

They are set up on bases in such a way that the acid mixer 1 is higher than the alkali mixer 2, to allow the kerosene to flow by gravity from the former to the latter.

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The process of acid treatment takes place in two steps: (1) first the preliminary drying is effected by the introduction of a small amount of acid (10 percent of the entire quantity), and (2) the main treatment then takes place.

The kerosene distillate, separated from water by settling, is pumped into the mixer 1. Then acid is supplied for preliminary drying from the acid egg (pneumatic lift) and the mixture agitated for half an hour by an air stream. Spent sulfuric acid is employed to conserve acid. The acid sludge takes 1-2 hours to settle. The acid sludge is then drained out, and fresh acid supplied to the mixer, without stopping the continuous agitation by the air stream. After agitation for 1-1.5 hour, the acid-distillate mixture is allowed to stand for 3-5 hours to settle, after which the acid sludge is drawn off into the acid sludge collector 4, while the distillate flows by gravity into the alkali mixer 2, into which an alkali solution of 5-10 degrees Beaume is delivered (under air agitation). As soon as litmus paper begins to turn red, the delivery of alkali solution is stopped. After 2-3 hours settling and draining off the alkali waste, a second portion of alkali is passed into the oil product to finally remove all harmful compounds.

After the second treatment, the alkali liquor contains considerable amounts of free alkali, and is therefore utilized for the first charge. The completeness of the purification from the petroleum acids is evaluated by the soda test, which is made as follows: 150 cubic centimeter of the kerosene and 9 cubic centimeter of a 2 degree Beaume caustic soda solution are heated to 70 degrees Centigrade in a flask; the mixture is then shaken

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and poured into a separating funnel, and the water extract is filtered into a test tube. If petroleum acids still remain in the kerosene still contains petroleum acids, they will form naphthenic soaps readily soluble in water.

On addition of a few drops of hydrochloric acid to the water extract, the naphthenic acid salts again turn into acids. Since the napthenic or petroleum acids are insoluble in water, the water becomes turbid. The quality of the treatment is thus judged by the degree of turbidity. 3 balls of treatment are distinguished: 1 ball indicating a transparent solution, 2 balls a slightly cloudy solution, and 3 balls a cloudy solution (only large print being legible through a test tube).

The Continuous Process Treatment of Kerosene

The batch treatment of kerosene in mixing tanks has the following disadvantages: airstream agitation is inefficient and therefore the acid is not fully utilized, and the kerosene is subject to oxidation by the airstream.

The continuous process of kerosene treatment using a mixer has proved to be more efficient. One of these plants includes 3 acid injectors and 4 settlers for acid, 3 alkali injectors and 4 settlers for alkali (Figure 140).

The acid is pumped successively through the injector 3, the settler 6, the injector 4, the settler 7, the injector 5, the settler 8 and finally enters the settler 16. The acid flows in the opposite direction contrary to the stream of kerosene (on the principle of countercurrent flow), i.e., fresh 95 percent sulfuric

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acid is delivered to the settler 8, where it meets the already treated kerosene so that the harmful impurities which are most difficult to remove can be taken out. The acid is drawn from the settler 8 into the injector 5, after which it again returns to the settler 8. The acid thus circulates for a few hours, until it is exhausted down to the strength established by the process chart. It is then transferred to the settler 7, and circulates as a stream of partially spent acid, between that settler and the injector 3, countercurrent to the fresh kerosene. The treated kerosene is discharged from the settler 8 into the settler 16 for final settlement to remove the acid sludge.

In mixing column 17 it is washed with water and pumped through 3 alkali injectors and 3 settlers for alkali.

The described principles of countercurrent flow are already being used in the alkali treatment. The spent alkali and acid kerosene meet in injector 13 and settler 10; in injector 14 and settler 11 -- partially spent alkali and incompletely refined kerosene; in injector 15 and settler 12 -- fresh alkali and refined kerosene (to completely draw off those harmful substances which are most difficult to remove). The final separation of kerosene from the alkali residues takes place in settler 18.

On treating the kerosene distillate the consumption of acid is 3 percent and of alkali, 0.004-0.006 percent of the distillate. The acid product is washed with water prior to the alkali treatment in order to reduce the consumption of alkali. Losses of product during treatment amount to 1-3 percent.

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Mixtures of 50 percent caustic soda and 50 percent soda ash are used in some cases in order to conserve the deficient caustic soda.

During the operation of the unit it is necessary to watch the strength of the acid being treated and to change it at the right time.

The alkali residues are removed from the unit only when the phenolphthalein paper indicates the absence of free acid.

It is very important to observe this process and to make sure the surrounding kerosene does not get into the treatment, since the water will dilute the acid and thus a large amount of it will be consumed.

lll. Treatment of Oil Distillates with Sulfuric Acid

The plants of the oil-refining industry produce more than 50 different types of oil. The viscosity of the oils varies from 1.3-1.4 at 50 degrees Centigrade to 7.0 at 100 degrees Centigrade.

Oil distillate contain the following harmful substances: tarry and asphaltic substances, sulfur compounds, naphthenic acids and unsaturated hydrocarbons. The main reason oil distillates are treated is to remove these harmful substances since they spoil the color of oils, promote the formation of scale coke, etc.

An acid-alkali oil unit, similar to the kerosene unit, consists of acid, alkali and washing mixers and driers.

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The acid treatment of oil distillates is carried out in 2-3 processes. The first batch of acid (20 percent of all the acid, which is required for treating the distillate) is provided for drying. All the remaining acid is spent in 1-2 processes on the basic acid treatment. The nature of acid treatment is as follows. The oil distillate, after the water and sludge has been removed, is pumped to the acid mixer 1. The acid descends by force of gravity in the required amount from the acid-measuring tank into the acid egg from which, under pressure, it is forced up into the acid mixer.

The oil is vigorously mixed with air for a better contact with the acid. Mixing of the distillate is carried out in 30-70 minutes depending on the capacity of the acid mixer: in small acid mixers this process takes less time, in large mixers it takes more. The distillates of many oils have a high viscosity at normal temperatures which makes mixing and, consequently, the treatment process as a whole very difficult. Therefore, before treatment the distillates are heated to the following temperatures:

Spindle-oil distillates	from 20 to 30 degrees C
Machine-oil distillates	from 25 to 40 degrees C
Cylinder-oil distillates	from 45 to 70 degrees C

A higher temperature during treatment is harmful, since the dissolving of the tars in the distillate is simultaneously increased and they are transferred from the acid sludge back to the distillate. Besides, hard and friable sludge is formed at an increased temperature which is very difficult to remove. After settling for 1-1½ hours the acid sludge is drained off

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into a container. After drying the acid sludge should be soft, ductile and black. If a greenish sludge is obtained, it means that it settled badly and that there is still some oil in it.

After drainage of the acid sludge, the main acid treatment of the distillate is conducted with the remaining sulfuric acid (80 percent of the whole batch). The acid is fed into the acid mixer with the vigorous mixing of the distillate with air. If the treatment process proceeds normally, then a considerable amount of the finest granules of acid sludge will be separated out. This may be seen on the glass if a little acid oil is deposited on it.

Toward the completion of the mixing, which usually lasts 2-3 hours, the fine particles flocculate. When the mixing stops, the flocculations of acid sludge begin to settle to the bottom.

To accelerate the settling of acid sludge, (1) the oil may be heated to reduce the viscosity (if there is a coil in the mixing tank), or (2), a small quantity of a precipitant, for instance, a caustic soda solution of 10 degrees Beaume, lube oil alkali or even water. The fine granules of acid sludge thereby rapidly coalesce to form large flocculations which rapidly settle to the bottom of the mixer. This phenomenon is termed coagulation.

Settling after treatment takes 2-4 hours. If the settling time is cut short, the remaining acid sludge may spoil the color of the oil and increase the consumption of alkali on further treatment. A sample on a glass slide may be examined to determine whether all the granules of acid sludge have settled. If the

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settling does not proceed well, additional coagulator must be added.

The drainage of the acid sludge terminates the acid treatment. The acid oil flows off by its own gravity to the alkali mixer 2 for neutralization.

The consumption of sulfuric acid in the treatment ranges from 2 to 6 percent of the distillate, depending on distillate quality.

112. The Alkali Treatment of Oil Distillates

The petroleum acids and acid sulfur compounds only pass over partially into the sludge during acid treatment, most of them remaining in the acid oil. The substances formed during the acid treatment also remain in the oil -- sulfonic acids, sulfuric acid esters and some amount of fulfuric acid itself -- and must all be neutralized by alkali.

The operating conditions for alkali treatment. The acid oil is slightly heated and a caustic soda solution added, with cautious agitation. The soda solution ranges from 3 to 5.5 degrees Baume. The more viscous the oil, the weaker the alkali must be: 5-5.5 degrees Baume is used for spindle oils, 3.5-4 degrees Baume for machine oils, and 3-5 degrees Baume for cylinder oils.

The choice of the alkali strength is very important. On treatment of a distillate with a weak alkali solution, the reaction of hydrolysis takes place simultaneously with the neutralization. This means that in the presence of water the salts

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of the naphthenic acids, otherwise called petroleum acids, that have been formed, may be again decomposed into acid and alkali. The more water there is in the mixer, i.e., the weaker the alkali solution, the stronger will be the hydrolysis. Stable oil emulsions may be formed on treatment of a distillate with strong alkali. Consequently, both weak and strong alkalis are harmful in the treatment of lube-oil distillates. The strength of the alkali solution for a given oil is therefore selected on the basis of trial and error. The alkali solution is added to the mixer, with continuous stirring, until the oil turns phenolphtalein paper pink, showing all slight excess of alkali. After 15-20 minutes of stirring the supply of air is stopped and the alkali liquor allowed to settle off from the oil. To accelerate the settling process the oils are heated: spindle oils to 50 degrees Centigrade, machine oils to 65 degrees Centigrade and cylinder oils to 70-80 degrees Centigrade.

The formation of emulsions. During the alkali treatment of lubricant-oil distillates, emulsions are rather frequently formed, and may be detected by the thickening of the oil, the appearance of small bubbles on the surface, and a change in its color. Low-viscosity oils (spindle, machine, transformer, turbine oils, etc) become white, while high-viscosity oils (for example cylinder oils) become brown or brownish-yellow. Failure of an oil sample taken during treatment to separate into layers when heated for 10-15 minutes on a heating bath indicates emulsification.

Emulsions are formed under the following conditions. On energetic agitation, oil and water are broken down into extremely

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minute particles. If the mixture contains sodium soaps of petroleum acids, the minute soap particles will surround the oil particles by a thin film and not allow them to coalesce on standing. A hydrophile emulsion (oil in water) is thereby formed. If resinous and asphaltic substances predominate in the mixture, they will surround the water particles while they themselves (these substances) remain dissolved in the oil and thus form a hydrophobe emulsion (water in oil). The emulsions formed in oil treatment are predominantly hydrophobe.

Only distilled water must be used to dissolve the alkali, since ordinary water contains various salts, including those of calcium and magnesium, which combine with the petroleum acids to form good emulsifiers.

To prevent emulsification, anti-emulsifiers are used, such as alkali liquor from kerosene treatment, canifol, acidol (petroleum acids). Kerosene treatment alkali liquor is the agent most frequently employed to break emulsions. They are added to the alkali solution in amounts ranging from 5 to 20 percent of such solution. Emulsification rarely occurs when this method of treatment is used, and the properties of the lubricant-oil alkali liquor is also improved (the oil content of such liquor being reduced).

Care must be taken that kerosene treatment alkali liquor used for this purpose does not contain kerosene, since this might lower the flash point of the finished oil.

The breaking of emulsions is a difficult task, although many methods are known: heating, addition of reagents (acids or

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alkalis), etc.

Only unstable emulsions can be broken by heating (to 70-80 degrees Centigrade), for stable ones are not broken even on prolonged heating at 90 degrees Centigrade. In such cases reagents must be added.

Before adding a reagent, the reaction of the emulsion -- acid or alkaline -- must first be learned. If the reaction is strongly alkaline, acid oil is added under cautions agitation until the reaction becomes neutral or faintly alkaline. If the emulsion is still not broken, still more acid oil is added so that the free sulfuric acid will break down the film of emulsifier. In extreme cases concentrated sulfuric acid is added, and this easily breaks the emulsion. But this step is very disadvantageous, since an additional quantity of acid must be used, and then more alkali to neutralize the additional acid as well.

If the reaction is acid, concentrated alkali is added to break the emulsion.

Gravity settling. On the completion of the alkali treatment, the oil is separated by settlement from the aquaeous solution of soap and sulfate, i.e., from the alkali liquor. The settling takes 4-6 hours, with the more viscous oils requiring a longer time.

113. Washing and Drying the Refined Oils

The neutralized oil still contains a certain amount of petroleum acids, which remain in it as a result of the hydrolysis of the soaps. A certain amount of the soaps formed during the

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alkali treatment is also left in it. The oil is washed with distilled water in alkali mixers or separate washing agitators to remove these substances. Usually two to three washings are given, using an amount of water equal to 15-20 percent of the amount of oil. The water and oil are heated to 40-70 degrees Centigrade (depending on the viscosity of the oil) and are then agitated for 45-50 minutes.

To save distilled water and alkali, the water from the third wash is reused for the second wash, and that from the second wash for the first wash.

After washing, the oil is allowed to stand and settle for 2-4 hours. The settled oil should not contain suspended soap granules, while the water should not have extracted any oil. The quality of the oil wash is judged by the soda test.

The soda test for oil is conducted differently than with kerosene. Arbtrary but equal quantities of oil and a 1 degree Beaume alkali solution are pured into a flask. The mixture is then strongly and rapidly agitated over a flame until it boils; it is boiled for 3 minutes and then placed on a water bath at a temperature of 80 degrees Centigrade. After settling, it is transferred to a test tube 15 millimeter in diameter. If the alkaline solution is as clear as water, the treatment is considered excellent and rated 1 ball. If the alkaline extract is turbid, but small print can be read through it, the treatment is considered good and rated 2 ball; if only large print can be read through it, the sample is considered satisfactory and rated 3 ball.

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The soda test determines the presence of petroleum acids in oil. The cloudier the sample, the higher the petroleum acid content of the oil. Washing must be continued until ball 2 is reached, bearing in mind that foreign substances may get into the oil during subsequent operations (storage, pumping) and lower the soda-test ratings.

After washing and settling, the oil contains moisture and must be dried. For this purpose the oil is pumped from the washing agitators into driers, heated to the necessary temperature, and blown with air. Spindle oil is heated to 50 degrees Centigrade during this operation, machine oil to 60-70 degrees Centigrade, and cylinder oil to 80-85 degrees Centigrade. To remove all the moisture, air must be blown through for 3-5 hours. The completion of the drying is determined by the transparency of the oil. If the oil is transparent, its treatment may be considered completed, and its agreement with the specifications is then tested in the laboratory.

A number of properties of oils are modified by the acid-alkali treatment and the resultant removal of harmful substances: specific gravity, viscosity, flash point, acidity, ash content, resin content, color.

TABLE 48

Oils	Reduction	
	in specific gravity	in viscosity, °E,
Spindle	by 0.001-0.002	0.1
Machine	by 0.002-0.004	0.2-0.4
Cylinder	by 0.005-0.006	1

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The higher the content of resinous and asphaltic substances in the distillate, the lower the specific gravity and viscosity of the finished oils.

The flash point of the distillates may be somewhat higher if they contain a certain amount of decomposition products. The removal of the petroleum acids from the distillates lowers the acidity of the oils to a level measured in thousandths.

The color of the finished oil is the factor which best characterizes the depth of treatment: the higher the color indices, the higher the quality of the treatment.

As will be seen from Table 49, the losses in sulfuric-acid treatment amount to 5-30 percent, depending on the distillate to be treated; they are 3-7 percent in the alkali treatment.

TABLE 49

	Losses	
Oils	in acid treatment	in alkali treatment
Spindle	5-6%	3-4%
Machine	8%	4%
Cylinder	8-10%	3-7%
Semi-tar	30%	---

114. The Leaching of Distillates. Utilization of Treatment Wastes

The solar oil and gas oil distillates consitute cracking stock or components for the manufacture of motor gasoline and require no treatment whatever. But it is just these destillates

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that contain the most petroleum acids. They are therefore leached to obtain these acids for utilization.

Leaching is performed in columns provided with steam coils for heating the product, and spray heats for spraying the leached distillates. Each column operates independently. A caustic soda solution of 3-5 - 5 degrees Beaume is charged in at a level a fifth of the column's height. The distillate is pumped into the bottom, and is sprayed through the spray head into tha alkali, slowly ascends in the column, and is already leached when it leaves the column. Its rate of passage through the column may be regulated in such a way that the reaction between the petroleum acids and the alkali is completely finished and no alkali is entrained with the distillate. In practice a column 2 meters in diameter and 8 meters high handles 100 tons of distillate a day.

The leaching process proceeds better at higher temperatures, and therefore the gas oil is heated to 60-70 degrees Centigrade, and the solar oil to 80-85 degrees Centigrade. The alkali liquor is regularly tested for free alkali by the laboratory. If a phenolphthalein test paper is tinged faintly pink, this means that the alkali is exhausted and fresh alkali must be substituted. For this the column is taken off stream, the alkali liquor is drained into a tank and the column is charged with fresh alkali. The leached distillate first passes to a settling tank for settling out the alkali liquor carried over, and then to a drier, where it is blown with air to free it of moisture.

The leaching of crude and mazuts. Sometimes the crude itself is leached. This facilitates the subsequent treatment of all distill-

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ates and reduces the corrosion of the equipment, since it removes the acid and active sulfur compounds from the crude. The production of low-grade alkali liquor is a disadvantage of the leaching of crude.

The total amount of petroleum-acid soaps and mineral oil carried over with them is termed the organic. The mineral oil content of the organic is:

in the kerosene wastes	6- 7%
in the gas-oil wastes	12-15%
in the solar-oil wastes	15-20%
in the lube-oil wastes	20-50%
in the crude-oil wastes	30-50%.

Crude oil alkali liquor cannot be utilized to prepare high-grade naphthenic soap or acidol, on account of their high content of mineral oils. At some refineries the leaching of crude and mazut is performed simultaneously with the process of distillation with soda ash, which is charged into the pipe still together with the mazut.

Acid sludges differ markedly from each other in composition and properties, according to the distillate treated.

Acid sludge from the treatment of light straight-run distillates is a black, mobile, tarry liquid. It contains up to 70-80 percent sulfuric acid and from 10 to 20 percent organic substances. The organic may be easily separated by diluting the sludge with water and heating to 60 degrees Centigrade.

Acid sludge from the treatment of cracked gasoline or the products of the pyrolysis of crude is a viscous liquid containing

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50-75 percent free sulfuric acid and 20-40 percent organic substances.

The acid sludges from the treatment of lubricating oils may be mobile or viscous black liquids, according to the distillate treated.

Sludges from the treatment of light lube-oil distillates contain up to 40-45 percent sulfuric acid and 45-55 percent organic substances. They are separated rather easily on dilution with water. Sludges from the treatment of machine and cylinder oil distillates contain 25-40 percent sulfuric acid and 45-60 percent organic substances.

For a long time no use could be found for the acid sludges, and therefore for many years the refineries disposed of them outside the city in great earthern sumps or pits, as a result of which over 500,000 tons of acid sludge in earthern sumps were accumulated at Baku alone. Such sludges contain 3-13 percent sulfuric acid and 85-95 percent organic mass.

Today we know many ways of utilizing acid sludges. Some of them are directed towards utilization of the acid, and others towards utilization of the organic components.

The sulfuric acid may be separated from acid sludge by steam treatment or dilution with distilled water at 60-80 degrees Centigrade. This liberates the free sulfuric acid in the following percentages:

Sludge from kerosene treatment	40-45% of 75-80% acid
Sludge from machine-oil distillate treatment	25-30% of 60-65% acid
Sludge from cylinder-oil distillate treatment	10-12% of 55-60% acid.

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The acid separated from acid sludge always has a dark color, but can still be used at treatment plants. Its evaporation produces a stronger sulfuric acid.

The organic mass separated from acid sludge may be used as stock for producing asphalt, as a fuel, etc. The same is done with fresh acid sludge. It is harder to use the sludge after it has been stored in earthern sumps, since it is an immobile and almost nonflowing mass, and it is very difficult to dig it out of the sump.

Alkali liquors contain soaps of the petroleum acids, sodium sulfate and free alkali.

The kerosene and gas-oil alkali liquors are the most valuable; they contain 15-20 percent soaps of the petroleum acids and 5 percent oils, and are good material for making naphthenic soap and acidol. They are also used as antiemulsifiers in the alkali treatment of lube oils. In the lube-oil alkali liquor the soap content is under 4-10 percent, and free alkali is 0.3 - 0.6 percent, both in terms of the liquor and the oil carried over makes up 30-60 percent of the organic. Some lube-oil alkali liquors can be used to produce naphthenic soap and acidol, as well as at the treatment plant itself in partial replacement of the free alkali.

115. The Disadvantages of the Acid-Alkali Treatment Method

The disadvantages of the acid-alkali treatment method are the high oil losses, the formation of an acid sludge which is difficult to utilize, the difficulty of alkali treatment and the

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high cost of the reagents.

In addition, the acid-alkali method cannot satisfy the increased demands made with respect to the characteristics of the oils (color, low viscosity, low solidification point, low resin content).

In general, high-grade oils cannot be produced by this method from distillates of heavy crudes: Binagia, heavy Balakhan, heavy Bibi-Eybat Crudes and others).

In due course new systems of treatment appeared -- with clays, with selective solvents, dewaxing, or removal of the paraffin wax, deasphalting, or removal of the asphaltic and resinous substances.

II. THE CLAY TREATMENT OF PETROLEUM DISTILLATES

116. The Refining Clays and Their Action

The refining clays consist mainly of silicon oxide (50-60 percent), aluminum oxide (10-30 percent), and a small quantity of the oxides of magnesium, iron and calcium. However, the principal factor in a refininf clay is not its chemical composition but its internal structure. Each particle of clay has an immense number of pores and minute channels, which make up a large interior surface, or, as it is called, porosity. The pores and channels are so small that they cannot be seen with the naked eye. A particle of clay magnified many times resembles an ordinary sponge. Thanks to the presence of pores and channels, clay possesses the property of absorbing (adsorbing) various substances out of solutions onto the surface of these pores.

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This capacity is termed adsorption, and substances capable of adsorbing are called adsorbents. The method of treatment by the aid of absorbent substances or adsorbents is termed adsorbent refining.

Different substances are absorbed to a different extent by an adsorbent. Resinous, asphaltic and other substances containing oxygen, sulfur compounds, and unsaturated hydrocarbons, are absorbed most easily. The aromatic hydrocarbons are absorbed with much more difficulty, while the naphthene and paraffin hydrocarbons are very poorly absorbed. For this reason, when oil products are mixed with refining clays, all the components of these products will not be adsorbed onto the surface of the clay, but mainly the resin, asphaltic substances, sulfur compounds, etc; while the paraffin, naphthene, and aromatic hydrocarbons will be almost entirely unadsorbed.

Chemical reactions also take place on the surface of the adsorbent, for instance, unsaturated hydrocarbons are not only adsorbed, but also polymerized.

Fairly numerous deposits of refining clays and earths are known. Those that are best known in this country are Gumbrin, extracted in Georgia in the village of Gumbri, and Zikeyevka earth (Zikeyevka Station, city of Zhizdra).

The refining clays and earths may be used in their natural condition or after activation, i.e., special treatment, washing and drying, which increases their absorptive power. Two methods of clay treatment of petroleum distillates are used at refineries:

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(1) percolation treatment, filtration of the distillates through clay in the form of granules;

(2) contact refining, treatment of the distillates with clay in the form of powder.

117. The Vapor-Phase Treatment of Cracked Gasoline

The vapor-phase treatment of cracked gasoline with clay is conducted in treatment towers at all two-coil Nefteproyekt-system cracking plants and at some Winkler-Koch plants. The treatment unit (Figure 143) consists of two towers, joined in parallel, and a supplementary rectifying column. 25 tons of granular Gumbrin or Zikeyevka clay is placed in each tower. The treatment unit operates as follows (Figure 93). The vapors of the cracked gasoline are directed immediately from the rectifying column of the plant into one of the treatment towers 2. After passing through the entire layer of clay from top to bottom, the treated gasoline vapors enter the additional column 3. With these vapors the polymers formed as a result of the treatment also enter this column 3 from the bottom of the treatment tower, together with the partly condensed gasoline. The gasoline vapors of the prescribed break-up are taken off overhead, while the polymers, which may be recracked, are collected from the bottom of the column.

The resinous substances are absorbed in the treatment towers, and polymers of the unstable diolefins are formed. The sulfur compounds contained in the distillate are removed by subsequent alkali treatment.

Each treatment tower operates continuously for one cycle, after which the clay must be regenerated by vigorous steam

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purging. A charge of clay operates up to 6 months without replacement.

The clay treatment of cracked gasoline has the following advantages over sulfuric-acid treatment:

Th unit is simple and forms a part of the entire gasoline-production process.

A considerable number of intermediate operations become superfluous: cooling and condensation of the pressure-distillate, pumping of the pressure distillate, storage in intermediate storage tanks; the consumption of scarce reagents is very small; the losses are insignificant.

A disadvantage of the process is the fact that the stability of clay-treated cracked gasoline is lower than that of sulfuric acid treated gasoline.

118. The Clay Treatment of Lubricants

To obtain lubricating oils of satisfactory characteristics by clay treatment of distillates alone would require the use of immense quantities of clay. For this reason the clay treatment of distillates is supplemented in practice by the acid-alkali or some other method of treatment to improve the color, odor and stability of the oil. In our refineries, the following operation sequence in the treatment of distillates has been adopted:

(1) leaching of the distillates;

(2) treatment of the leached distillate with sulfuric acid to remove most of the resins;

(3) treatment of the acid oil with clays.

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A description of the flow sheet of one of the oil-treatment plants (Figure 144) is given below.

The oil, after being freed from petroleum acids, treated with acid, and separated by settling from the tar residue, enters the large mixer where it is agitated by the help of a turbo-agitator (a vertical shaft with turbine wheels) with ground Gumbrin which is delivered by pump 14, specially adapted for this operation. The mixture of acid oil and Gumbrin is pumped by a steam pump through pipe still 5, where it is heated to 230-280 degrees Centigrade (usually to 30-40 degrees above the flash point of the oil). The heated mixture from the pipe still enters the evaporator 6, at the bottom of which steam is also introduced to strip the oil of light ends and decomposition products. The final clay contacting of the oil takes place in column 7 by the aid of pumped circulation. To separate the clay from the oil, the mixture, heated to 120-140 degrees Centigrade, is pumped through a filter-press. The filter-press (Figure 145) consists of 36 discs, over which iron screening with large openings (about 2.5 millimeter is stretched; a very fine copper mesh is stretched across the iron screen. The mixture of oil and clay is delivered to the inside surface of the discs between the screens. Under the pressure of a pump, the oil passes through the iron net then through the copper net, while most of the clay is retained by them. For the final separation of the clay, the oil is pumped through the frame filter-press 13; filter paper or filter cloth is stretched across the discs in the filter-press. The filtered oil then flows by gravity into storage, while the spent clay is removed from the plant and dumped into pits.

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The preparation of clay for the clay treatment of lubricating oils is very important. The clay must be in a finely divided (powder) state and must also be entirely dry. The clay is prepared at a unit called a mill. This unit consists of a crusher, a conveyor, the mill itself, silos, and fan and other equipment. The clay is first broken in the crusher into lumps 1-2 inches in size. The crusher is the usual type of stone crusher. The clay is delivered by a conveyor from the crusher to the hopper of the mill. The mill has 4 millstones with rollers, which grind the clay to a fine powder as they rotate. The clay is then dried and collected in silo-storage bins.

119. The Percolation Treatment of Lubrication Oils

Percolation treatment (or percolation filtration) is used with oils that have already undergone acid-alkali, contact or selective treatment.

This method is used only in cases where it is necessary to improve the color of the oil. The filtration takes place in cylindrical towers, on the top of which a layer of granules is scattered (Figure 146). At the bottom of the tower, to avoid loss of clay, a filter cloth is stretched.

The oil heated to 150 degrees Centigrade is delivered to one of the towers. It then passes all the layers of clay granules and flows by gravity into storage. The first portions of the oils passing through fresh clay granules, are almost completely purged of tar. As the clay becomes fouled, the color of the oil deteriorates, and finally the oil leaves the tower almost unchanged in color. For this reason the treatment is shifted

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to the other tower while the color of the oil is still within the limits set by the norms.

To extract the oil from the clay in the tower (Figure 146) ligroin is introduced as a solvent. The flushing with ligroin is continued until it emerges from the tower with a light yellow color. The ligroin is then purged from the clay by steam for 4-7 hours, after which the tower is again ready for operation. The solution of oil in ligroin is passed into a still, in which the ligroin is separated from the oil. The ligroin is used for further flushing, while the oil is used as a second-grade product.

About onee a month the clay is regenerated, for which purpose it is charged into a special furnace, where it is sintered by hot gases at a temperature of over 500 degrees Centigrade.

III. TREATMENT BY SELECTIVE SOLVENTS (SELECTIVE REFINING)

120. The Significance of Selective Refining and its Essential Principles

The above described methods of treatment have the following fundamental disadvantages: high losses of oil, production of wastes that are difficult to utilize (acid sludge, spent clay), the impossibility of producing high-grade oils from heavy tarry crudes, and the high consumption of reagents in short supply.

Although clay treatment has a number of advantages over acid-alkali treatment, it still cannot produce high-grade oils.

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A more improved method of treatment of oils is treatment by selective solvents, for instance, furfurol, phenol, a mixture of propane and cresol, etc.

By this method the resources of oil stock are increased on account of the possibility of processing heavy resinous crudes. Another advantage is that the oils produced are of high quality (excellent color, high index of viscosity, low coking number, increased chemical stability) and the waste products may be completely utilized.

So that selective treatment may be understood, the following illustration is presented. We take a solution of 200 cubic centimeter of alcohol in 1 liter of kerosene and mix it with 1 liter of water. The kerosene does not dissolve in water. The alcohol dissolves considerably better in the water than it does in the kerosene. For this reason, when the solution is mixed with water, the alcohol is separated from the kerosene and dissolved in the water. As a result we obtain 1 liter of kerosene and 1.2 liters of an alcohol-water solution. This method of extracting a dissolved substance from one solvent by the aid of another is termed extraction. This property of some liquids of dissolving some substances and not dissolving others is utilized in the treatment of lubricant distillates. To obtain a high-grade oil the distillate must be separated into two parts: one of them is called, by general agreement, the paraffinic portion while the other is called the napthenic portion.

The paraffin hydrocarbons belong to the paraffinic portion, together with the napthenic and aromatic hydrocarbons with long

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chains. This part of the oil is chemically stable and does not produce resinous residues or carbon deposits. The paraffinic portion represents purified oil and is by general agreement called raffinate.

The resinous and asphaltic substances belong to the naphthenic portion, together with the polycyclic aromatic and naphthenic hydrocarbons with short side chains. This portion possesses directly opposite properties: it is chemically unstable, gives carbon deposits and deposits tars. The solvent with the naphthenic portion dissolved in it is called the extract solution, while the napthenic portion itself is called the extract.

121. A Furfurol Selective Treatment Plant

Furfurol may be prepared from straw, sawdust, sunflower-seed husks, corncobs, etc, on heating with sulfuric acid. This solvent is therefore cheap and widely used. However, although it gives excellent results in treating distillate oils, it does not do so when used on residual oils.

A a plant for treating oils with furfurol, the following processes take place: (1) mixture of the solvent with the distillate and its separation into two parts; (2) distilling the furfurol off from the raffinate; (3) distilling the furfurol off from the extract; (4) fractionation of the furfurol. The first process is simple and is effected in an ordinary packed column. All the remaining processes -- the separation of furfurol and its regeneration -- require rather complicated equipment.

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The flow sheet for a furfurol plant is as follows (Figure 147). The distillate, preheated in the steam preheater 3 to 50-70 degrees Centigrade, is introduced into the bottom of the packed extracting column 5, while furfurol, preheated in the steam preheater 4 to 60-100 degrees Centigrade, is introduced from the top. The furfurol and distillate flow countercurrent in the column and mix. From the top of the column the raffinate with a small amount of furfurol is taken off, while the solution of spent oil or furfurol extract is discharged at the bottom of the column. The raffinate is then preheated in the furnace 9 and delivered to the rectifying column 10 to drive off the furfurol. In the stripping column 12 the raffinate is finally freed from the last furfurol, and is then pumped by the pump 42 into the finished-oil tank. The watery furfurol is taken off overhead from the rectifying column and the stripping column and is collected into the receiver 28.

The extract is collected from the tank 7 by the pump 14, pumped through the pipe still 15 and delivered to the atmospheric column 16 to drive off the furfurol. Only a part of the furfurol is eliminated here. It is not possible to eliminate all of it in an atmospheric column, since the extract contains up to 90 percent furfurol. The temperature of the extract also cannot be increased up to the complete evaporation of the furfurol or else the furfurol will be decomposed and resinified. For this reason, the principal separation of furfurol from the extract is effected in the vacuum column 18. To maintain the necessary temperature in the column, the extract is forced to circulate from the bottom of the column through the pipe still 15 and back into the column.

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from the circulating product, the excess portion of the products is continuously pumped by the pump 26 into the stripping column 23, where the residual furfurol is driven off with live steam. The extract is then pumped by the pump 35 into a receptacle.

The last operation is the restoration of the furfurol. A portion of the furfurol is freed from water during the course of the process and is collected in the receivers 21 and reused. The remainder of the watery furfurol is collected in the receivers 28 and 34, from which it is delivered to the furfurol rectifying column 38 to remove the water; it is then also collected in the receiver 41 for reuse.

We present the results of furfurol treatment of one of the distillates.

1.93 parts of furfurol by volume is taken to each part of distillate. After processing under the following conditions,

Temperature at top of column	113° C
Temperature at bottom of column	100° C
Yield of finished oil	75.5%

the qualities of the distillate are changed and are then characterized by the following indices:

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TABLE 50

	Distillate	Raffinate	Extract
Specific gravity	0.915	0.84	0.995
Flash point, ° C	260	266	--
Viscosity at 98° C, Engler °	2.66	2.22	7.45
Coke content by Conradson method	1.4	0.14	--
Index of viscosity	64	97	--

The extract is used as a fuel.

122. Treatment of Oils by a Double-Solvent (Duosol Process)

The method of oil treatment by two mutually insoluble solvents, for instance, propane and cresol, is also frequently used. Propane does not dissolve resinous and asphaltic substances but is an excellent solvent for paraffin and naphthene hydrocarbons. Cresol dissolves resinous and asphaltic substances and unsaturated hydrocarbons, but does not dissolve the remainder of the distillate. On combined treatment with propane and cresol, two products are obtained: a solution of the oil itself in propane, and a solution of the extract in cresol.

The method of treatment with two solvents is the most widely used process of oil treatment at the present time. This process has solved the very difficult problem of treating the high-resin residues from the straight-run distillation of crude, to produce high-grade oils.

The reagent used in this process is a mixture of technical propane with a mixture of solvents. The latter consist of phenol and cresol, with mixtures Nos 0, 1, 2, and 3 being distinguished, depending on the phenol content of the mixture. In the USSR,

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mixture No 3, which consists of 36-38 percent phenol and 64-62 percent cresol, is used. The equipment of the treatment plant is divided into an extracting and a regenerating section and consists of extractors, evaporators, atmospheric and vacuum column, exchangers, coolers and other units.

THE FLOW CHART (Figure 148). The process is conducted in a countercurrent extraction system consisting of 9 mixing and settling sections: 4 sections in extractor E-1 and 5 sections in extractor E-2. The liquid propane is introduced from reservoir P-5 into section 1, the phenol-cresol from reservoir N-5 into section 9, and the feed from the mixer 10 into section 3. There are two layers in each section, the upper one of raffinate with propane, and the lower one of extract with phenol-cresol. There is a device built into the section baffles allowing a product to be drawn from one section into another (see Figure 148); and each section is provided with an individual section pump for circulating the liquid from one section to another.

For instance, the lower layer of section 2 is collected by a pump and delivered together with fresh propane to section 1. The upper raffinate layer is thereby enriched by useful hydrocarbons, while the lower, or extract layer, is enriched by asphaltic and resinous substances. At the same time, the lower layer of section 3 is collected by another pump and is pumped through the first section into the second. At the same time the raffinate layer, enriched in propane, is injected from section 1 into section 2. The useful hydrocarbons pass from the extractor section 3 into the upper layer, while the asphaltic and resinous substances pass from the raffinate layer into the lower layer. An exchange, as it

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were, of useful and harmful substances takes place between the extract and the raffinate. The extract of section 4 and the raffinate of section 2 enter the mixer 10, where they are mixed with fresh feed stock. The mixture passes from the mixer 10 through section 2 into section 3. At the same time, a certain amount of raffinate with propane is drawn from section 2 into section 3. There, also, the feed becomes stratified into raffinate and extract.

From section 5, the extract is delivered through section 3 into section 4, etc. A fresh mixture of the phenol and cresol solvents enters section 9 and moves from section 9 to section 1 becoming more and more enriched with harmful substances on the way, while fresh feed moves from section 3 into section 9 countercurrent to the phenol-cresol mixture and becomes more and more enriched with the useful hydrocarbons on the way.

The raffinate layer from the upper part of section 9 and the extract layer from the lower part of section 1 are routed into the corresponding regenerating system.

The regeneration of solvent from the extract. The mixture containing about 20 percent propane, 74 percent phenol-cresol and 6 percent oil, passes from lower part of section 1 to the evaporator N-1, operating under 13 atmospheres pressure. Before it enters the evaporators, the mixture is divided into two streams: the first enters the evaporator at the same temperature at which it leaves the extractor (28-29 degrees Centigrade), while the second stream is heated in the exchanger 11 by the heat of the hot phenol-cresol, coming from the evaporator W-1.

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In the evaporator N-1, the temperature is held at about 66 degrees Centigrade. The evaporated propane is liquefied in the condenser 12 and enters the propane tank P-5. In the evaporator N-1, up to 86 percent of the propane is removed. Another 12.5 percent of propane is then taken off in the additional evaporator N-2 as a result of reducing the pressure to 3 atmospheres, and, after liquefaction, is also routed into the tank N-5. The mixture of phenol-cresol with oil is then heated to 166 degrees Centigrade in the exchanger 14, and enters the column N-3. The mixture is here heated by steam to the required temperature. The phenol-cresol vapors leaving column N-3 pass through the exchanger 16 and then enter the evaporator W-1.

From the lower part of the column N-3, the product is pumped into the column N-4, operating under a vacuum of 40 millimeter mercury at 316 degrees Centigrade. The heat necessary to heat the product to this temperature is supplied by the circulation from the column into the pipe still 17 and back into the column.

The phenol-cresol vapors leave the upper section of column N-4; they are then condensed (18), cooled (19) and passed to the receiver 20. This product is utilized as a reflux for columns N-3 and N-4. The extract is discharged from the lower section of column N-4 at 274 degrees Centigrade, cooled and then pumped into the cooler 21. Not more than 0.05 percent of phenol-cresol still remains in the extract.

The regeneration of the solvent from the raffinate is effected in evaporator P-1, the additional evaporator P-2, column

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P-3 and vacuum column P-4. The flow sheet is similar to that we have just described for the regeneration of the solvent from the extract.

Propane is taken off from the upper section of the evaporator and goes to storage. The raffinate from the lower section of the evaporator P-2 is discharged into the intermediate collector E-3, where the extract carried over with the raffinate is separated and returned to sections 6, 7 and 8 of extractor E-2. Columns P-3 and P-4 operate in the same way as columns N-3 and N-2. The necessary heat for the operation of column P-4 is supplied by the heating of the raffinate circulating through the second half of the furnace 17. The raffinate, containing not more than 0.1 percent phenol-cresol, is discharged from the lower section of column P-4.

We have mentioned evaporator W-1 above. It operates under 0.25 atmosphere pressure at a temperature of 102 degrees Centigrade overhead and 182 degrees Centigrade on the bottom. The product entering evaporator W-1, consists of phenol-cresol, water and propane. The dehydrated phenol-cresol is eliminated from the lower section of evaporator W-1, while the dehydrated propane is taken from the upper section of the small evaporator W-2.

As is clear from the description, the flow sheet of the double-solvent refining plant is fairly complicated. For this reason it is completely automatized and is provided with a large number of control instruments.

The yields and characteristics of the product. An approximate material balance of the treatment of tars from a paraffinic crude

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is given below

Taken:	Using a low-resin stock,%	Using a high-resin stock, %
Concentrate	100	100
Propane	300	300
Phenol-cresol	500	500
Obtained:		
Raffinate	60	41
Extract	390	58
Propane	299.5	299.5
Phenol-cresol	499.7	499.7
Losses:		
Oil	1.0	1.0
Propane	0.5	1.5
Phenol-cresol	0.3	0.3

Thus: (1) The ratio between phenol-cresol, propane and concentrate is 5:3:1; (2) the losses of solvent are very small (0.5 percent propane and 0.3 percent phenol-cresol); (3) the yield of raffinate from the stock was 41 percent in the first case and 60 percent in the second.

We present below the characteristics of the stock and the oils produced by double-solvent refining.

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TABLE 51

Characteristics	Stock	Raffinate	Extract
Specific gravity	0.927	0.903	1.075
Viscosity at 100° C	5.84	3.68	--
Index of viscosity	--	78.5	--
Yield, %	--	85.7	--

123. Conditions of the Selective Refining of Oils

The following fundamental conditions affect the process of selective refining of oils: quantity and quality of the solvent, temperature and methods of treatment.

The characteristics of the solvent are the basic factor affecting all the remaining conditions of the process. It is determined by two properties:

(1) The capacity to sharply separate stock into useful and undesirable substances and to form two layers: a solution of extract, and a raffinate; this property is called the selective capacity (selectivity) of a solvent;

(2) the low solubility of the oil to be refined; this property is termed the solvent power of a solvent. As the solvent power increases the yield of oil diminishes. Ordinarily, a solvent with a good solvent power has a poor selectivity anc vice versa. Nitrobenzene belongs to the first group of solvents, and furfurol belongs to the second.

These properties depend largely on the temperature, i.e., the higher the temperature, the more soluble oil is in the solvent.

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A solvent must also possess the following properties: it must be easily and completely regenerated, it must not react chemically with the refined oil and it must not have toxic or corrosive properties. At first nitrobenzene was used for selective refining, but its use under refinery conditions is limited. This is explained by the fact that nitrobenzene is highly toxic and has a high boiling point (211° Centigrade), which is inconvenient for regeneration.

At the present time the most widely used solvents are furfurol, propane with phenol-cresol, phenol, chlorex, sulfur dioxide and benzol. We present below data on the world distribution, on 1 January 1943, of the various selective processes (in percent):

Double-solvent	30.0
Furfurol	24.5
Phenol	24.5
SO_2 + benzol	10.5
Chlorex	6.0
Nitrobenzene	4.5
Total	100

An increase in the quantity of solvent in selective refining improves the quality of the oil but tends to reduce the yield. For instance, in refining distillate with an equal volume of nitrobenzene, the yield of oil with an index of viscosity of 99.5 is 61 percent; if two parts by volume of nitrobenzene are used, the yield of oil is reduced to 48 percent, but the index of viscosity rises to 101; finally, when the oil is treated with 3 parts of the solvent by volume, the yield of oil

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amounts to only 35 percent with an index of viscosity of 105. At the same time a number of other characteristics of the oil are improved.

In selective refining, the consumption of solvent depends to a considerable extent on the properties of the stock and of the solvent itself.

The influence of temperature on refining. When the treatment temperature is raised, oil dissolves better in any solvent; thus, on account of an increased temperature the consumption of the solvent may be reduced. For instance, when oil is refined by an equal volume of nitrobenzene at 20 degrees Centigrade, the yield is 64.5 percent, but at 40 degrees Centigrade it is only 52.4 percent. However, the treatment temperature cannot be raised indefinitely: a moment finally arrives when the oil is completely dissolved in the solvent and the purpose of the solvent, namely, the selective extraction of the harmful substances, is entirely lost. For this reason such optimum condition are chosen as will assure the highest quality of the raffinate with the maximum yield of it. For instance, it may be seen from the data presented below that when oil is refined with a mixture of aniline and benzol, we must stop at a temperature of 23 degrees Centigrade, since at any higher temperature the quality of the oil is reduced along with the yield.

Method of refining and choice of the optimum conditions. The quantity of solvent and the treatment temperature are selected so that the purification of the distillate from harmful substances is complete, and the optimum yield of oil obtained. These conditions are in the first instance selected experimentally. But the indices

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of the refining depends not only on the conditions but also on the method of refining. There are a number of methods for refining lubricants: cocurrent, countercurrent, with recycling, under variable temperature conditions.

In the cocurrent method, the stock is treated all at once by the entire quantity of solvent. The two layers formed thereby are separated in the settling tank, and the solvent distilled off from each of them. This is the most disadvantagous method of all and is seldom used.

In the countercurrent method, the stock and the solvent move countercurrent to each other (Figure 149). The refining may be effected in various apparatus. If it is done in columns filled with a packing of clay rings, then the heaviest part (for instance, furfurol) is delivered at the top while the lightest part (for instance, the stock) is delivered at the bottom; the lightest fractions of the stock ascend and, when they come in contact with the fractions of the solvent, give up their undesirable substances. The raffinate is taken off overhead, while the solution of extract is taken off at the bottom. An extraction in stages is conducted in the mixing tanks and settling tanks as it is in the double-solvent method.

The countercurrent method consumes only one half to one third as much solvent as the cocurrent method.

Countercurrent extraction may be done with recirculation or under variable temperature conditions.

Operation with recirculation. The stock is processed at

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higher temperatures, when the solvent power of the solvent is high and its selective power, on the contrary, is low. The undesirable substances are thereby completely eliminated from the stock, and at the same time a considerable part of the raffinate is also extracted. The solution extract is then cooled, thus increasing the selective power of the solvent and reducing its solving power; the raffinate with which a certain amount of undesirable substances was carried over is thereby separated from the extract solution. The raffinate so separated is returned for re-extraction. By this method, the consumption of solvent is diminished and a high yield of high-grade oil is achieved.

This method is used, as we have seen, in the double-solvent refining method.

The method of refining under variable temperature conditions consists in maintaining a high temperature at the place from which the raffinate is drawn off, and a low temperature at the place from which the extract is drawn off.

The essential features of this method consist in: (a) separating from the stock, at reduced extraction temperatures, most of the undesirable substances, part of which still remains in the stock owing to the low solvent power of the solvent at the given temperature; (b) treating the raffinate with solvent at a higher temperature, thereby extracting the rest of the undesirable substances from it.

This method is employed in the furfurol process described above.

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The normal conduct of the operating conditions at the plant should assure: (a) the production of oil of the required characteristics, (b) maximum yield of oil and (c) economy in solvent consumption.

As has been already stated, the quality of the oil depends on the temperature of refining and on the quantity of the solvents. The quality of the finished oil is regularly tested in the laboratory. If the quality of the oil should deteriorate, the temperature must be raised, or, if the refining temperature is normal, the flow of solvents must be increased. The oil may be over-refined; in that case it is necessary to lower the temperature to normal or reduce the flow of solvent. Changes in the operating conditions are made gradually, and it is necessary to test the quality of the oil in the laboratory after each such change.

The maximum yield of oil also depends on the normal operating conditions of refining: on the refining temperature and the quantity of solvent.

It is well known that on raising the temperature of refining and increasing the flow of solvent, the quality of the oil improves more slowly, while the yield of oil falls more rapidly. For this reason deep refining must not be abused.

Water, which may form an emulsion in the extract, affects the yield of oil. The presence of this water leads to entrainment of the oil when the solvent is driven off, and to consequent oil losses. It is therefore necessary to make sure that watery oil or watery solvents do not enter the refining system.

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Solvent losses depend on the conduct of the regeneration process, the stripping of the solvent from the raffinate and extract, and on its dehydration. The stripping of the solvent is checked by the laboratory according to the quality of the oil, and in practice the solvent should be driven off completely. The process of separating the water from the solvent and that of dehydrating the solvent must also be watched carefully.

IV. THE DEWAXING AND DEASPHALTING OF LUBRICATED OILS

The methods of treatment of distillates that we have just considered allow the production of lube oils only from what is termed oil stock, i.e., from mazut with only low content of paraffin and asphaltic and resinous substances.

Distillates with high paraffin content, and consequently with a high freezing point, are obtained from paraffinic mazut. It was formerly thought that paraffinic mazut could not be used for lube oil stock, and it therefore was used mostly for cracking. However, it turned out that after the removal of paraffin from a paraffinic mazut, high-grade oils could be produced with a high index of viscosity. For this purpose the so-called dewaxed oils are used.

The solid paraffins are contained in the lube-oil fractions of paraffinic crudes and are responsible for their high freezing point.

There are two types of solid paraffins: the paraffins proper constisting of large crystals, and the ceresins consisting

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of small crystals. The paraffins consist of large lamellar- or band-shaped crystals, while the ceresins consist of very tiny acicular-shaped crystals. The paraffins, having a relatively low molecular weight (300 to 500), and a boiling point between 370 and 560 degrees Centigrade, can be distilled over with the lube-oil distillates. The ceresins have a higher molecular weight (520 to 750) and a boiling point of 500 to 700 degrees Centigrade; they are contained in semi-tars and tars and cannot be distilled without decomposition.

The ordinary commercial paraffins have a melting point of 40 to 56 degrees Centigrade, which for the ceresins is from 65 to 85 degrees.

Paraffins are produced from paraffinic crudes at special plants. The ceresins are obtained from ozocerite or montan wax. There are deposits of ozocerite on Cheleken island in western Ukraine. Ceresins are also obtained from the residues that settle out when crudes are stored in tanks, on pumping them into pipelines, and in their wastes from the production of aviation oils and petroleum.

In some cases (for instance, from Groznily paraffinic crude), the paraffin may be removed from the distillate by the following method. The paraffinic distillate is cooled in a "pipe in pipe" cooler to 2 to 6 degrees Centigrade. The large crystals of paraffin formed are filtered off on frame filter-presses. The filtered distillate has a freezing point from + 8 degrees Centigrade to + 15 degrees Centigrade. The paraffinic distillate is then recooled to a lower temperature of - 15 degrees Centigrade.

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Most viscous oils contain small-crystal ceresin, which cannot be separated by this method.

In dewaxing high-viscosity oils, the following operations are performed: (a) the distillate is dissolved in a specially selected solvent; (b) the solution is heated until the paraffin is completely dissolved; (c) the solution is slowly cooled to a low temperature; (d) the paraffin precipitated out on cooling is removed on filter-presses or by centrifuges; (e) the solvent is distilled off from the oil in a distilling plant.

Distillates are unsuitable for the production of high-viscosity lube oils, and therefore the residues from the distillation of mazut -- semi-tars and tars -- must be used. Most of the asphaltic and resinous substances are concentrated in these residues, and their separation by the processes known to us is very difficult. For this purpose the method of deasphalting is used, i.e., the removal of asphaltic substances by treating the distillate or residue with liquefied propane.

124. Naphtha Solvent Dewaxing

For dewaxing oils, solvents are also used which readily dissolve oil, but only sparingly dissolve solid paraffins: liquid propane, a mixture of benzol with acetone and toluol, a mixture of methyl-ethyl-ketone (MEK) with benzol and toluol, a mixture of dichlorethane with benzol (Barisol), the naphtha-fraction of gasoline boiling at 100 to 149 degrees Centigrade.

The dewaxing of refined oil in naphtha solution to remove the paraffin and ceresin from it, to obtain aviation oil, is practiced at one of our refineries.

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The flow sheet (Figure 150). The refined oil and naphtha is delivered into the orifice mixer 1 in the proportion of 2 to 3 parts naphtha by volume to each part oil by volume. The mixture is preheated to 40 degrees Centigrade in the preheater 2 and is dehydrated, after which it enters the tank with the propeller-agitator 3. The propeller keeps the liquid in motion so that the ceresin does not settle out. The mixture is then forced into the 23 cooling towers 4, which consist of vertical columns in which coils of 100 2-inch pipes are set. The mixture passes through the shell space while a cooling solution of ammonia passes through the tubes. The mixture is cooled at a rate not over 1.5-2 degrees Centigrade per hour, and, when cooled to - 40 degrees Centigrade, enters the pressure feed tank 5, from which it is delivered to the centrifuge 6 (Figure 151). In the centrifuge, revolving at 17,000 revolutions per minute, about 18 percent petroleum is removed.

In removal of petrolatum from oil (Figure 152) is based on the difference between the specific gravities (1) of the solutions of petrolatum (0.816) and oil (0.780) and is accomplished by the action of the centrifugal force (2), developed by the rotation of the centrifuge. During this process, the heaviest fractions of the petrolatum solution (3) are thrown off to the edge of the centrifuge, while the oil fractions of the solution (4) are concentrated around its axis. The solution of oil in naphtha, and the solution of petrolatum in naphtha, are then collected in the special receivers 9 (see Figure 150). The oil is then cooled to - 29 degrees Centigrade in the cooler 10, and is pumped through the first 17 towers to regenerate the cold. The oil solution then enters the tank 12, from which it is routed to the

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distilling unit to remove the naphtha, which is recycled, while the oil is routed into a vacuum drier to remove the remaining moisture and then into the commercial storage tanks. Below is an approximate material balance of the process:

Taken:	
Refined oil	100 tons
Naphtha	235 tons
Total	335 tons
Produced:	
Finished oil	68 tons or 68 %
Petrolatum	31 tons or 31 %
Naphtha	225 tons
Losses:	
Naphtha	10 tons or 4.5 %
Oil	1 ton or 1.0 %
Total	335 tons

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TABLE 53

	Specific gravity	Viscosity at 100° C	Freezing point, °C	Coke in %
Original crude oil	0.918	4.5	+ 38	+
Refined oil	0.892	3.01	+ 52	+
Mixture with naphtha before centrifugation	0.785	--	+	+
Dewaxed oil solution	0.778	--	+	+
Petrolatum solution	0.816	--	+	+
Finished oil	0.908	3.35	- 14	0.56
Petrolatum	0.875	2.64	--	--

The petrolatum contains up to 25 to 30 percent oil.

This disadvantage of the process of dewaxing in the presence of naphtha:

1. The differential between the dewaxing temperature and the solidification point of the dewaxed oil reaches 20 to 30 degrees Centigrade. This requires the dewaxing to be performed at very low temperatures.

2. The rate of lowering of the temperature of the solution does not exceed 1.5 to 2 degrees per hour which requires bulky equipment and involves high losses of cold.

3. The high losses of naphtha (up to 10 percent of the amount of the oil stock).

4. A large amount of oil is carried over with the petrolatum and lost.

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125. MEK Dewaxing

Dewaxing with methyl-ethyl-ketone, or MEK, is the most widely employed method of dewaxing oils at the present time. The solubility of ceresins and paraffins in MEK is very low as a result of which MEK is an excellent reagent for the process of removing paraffin; but on the other hand, MEK is not a sufficiently good solvent for oil, so that, during dewaxing part of the oil will be precipitated along with the paraffins and ceresins.

Benzol and toluol are usually added to MEK. These substances are readily soluble in MEK and increase the solubility of oils in it. Usually a mixture of 25 to 40 percent MEK, 15 to 25 percent toluol and 40 to 60 percent benzol is used.

A MEK dewaxing plant consists of the following principal equipment: exchangers, coolers, evaporators, rectifying columns and separators.

Flow chart (Figure 153). The feed stock -- the oil 1 -- is mixed with the solvent 25 in continuous process mixers, with the delivery of solvent and feed regulated in the ratio of 4:1.

Before the mixture is routed to the dewaxing plant, it is preheated to convert all the solid paraffins and ceresins into the liquid state. For this purpose it is pumped through the steam preheater 2, where the mixture is heated to 50 to 70 degrees Centigrade by the exhaust steam 42.

To cool the mixture to -30 to -35 degrees Centigrade, it is passed through a cooler system: the water coolers 3 where it is cooled by circulating water, through the exchangers 4 where it is

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further cooled (to -4 to -5 degrees Centigrade) by circulating filter oil (from 8 to 18) [sic] and, finally, through the coolers 5 where the solution is cooled by circulating amonia to -30 to -35 degrees Centigrade.

The cooling mixture is delivered to the supply tank 6 and then to the vacuum filters 7 for filtration.

The vacuum filters consist of a rotating drum across which a filter cloth is stretched, submerged in a steel shell, together with a special distributing system. The drum rotates very slowly (at a speed of 10 to 30 revolutions per hour).

The cooled mixture is delivered to the vacuum filters, where it is freed from petrolatum and flows by gravity into the receiver 8. The cake of cheese paraffin formed is washed away by fresh solvent and removed to the settling tank 11 by the screw conveyer 9.

To facilitate the removal of the cheese paraffin from the filter, flue gases are passed into the filter after first being cooled by ammonia in the cooler 12, The flue gas (15) enters the plant through the gasholder 14. The vacuum pump 13 collects the gas from the gasholder and pumps it through the cooler into the filters. The used flue gas is recycled to the system.

The filtered oil, as has been said already, circulates through the exchanger 4, where it is heated, and then passes to the filtrate evaporater 19. The cheese paraffin in the form of a slurry passes to the upper plate of the evaporator 20 for recovery.

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The evaporators are equipped with plates having dead-end steam coils. The filtrate and petrolatum flow from all plates down to the bottom of the evaporators. Almost 98 percent of the solvent is vaporized during this process and is routed through the coolers 21 and 22 into the solvent tank 23, from which it is again recycled into the system.

The remaining solvent is driven off in the filtrate-stripping column 27 and in the petrolatum-stripping column 31, through the bottoms of which superheated steam is passed.

The solvent evaporated in the stripping columns, together with the steam 33, enters the cooler 34, and then passes to the separator 35 for removal of water. From the top of the separator the dehydrated solvent is passed through the line 36 into the solvent tank. From the lower section of the separator, the condensate together with the solvent is passed through the line 37 into the rectifying column 39. The condensate with the solvent from the settling tank 11 also enter this column, in which the solvent, containing (in the condensate) up to 10 percent water, is driven off. Since the quantity of the solvent stripped is small in comparison with its total mass, it is directed by line 36 into the solvent tank without further dehydration.

As a result of the recovery for circulation, 99.8 percent of the solvent is returned. Below are indicated the results of dewaxing a semi-tar from a light paraffinic crude by MEK and benzol (60 percent and 40 percent respectively) at -35 degrees Centigrade and -23 degrees.

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The achievements of this process. The MEK dewaxing process is distinguished from the naphtha solution dewaxing method:

(1) by the small temperature difference between the process temperature and the temperature of oil and the dewaxed oil -- 5 to 8 degrees Centigrade instead of 20 to 30 degrees Centigrade;

(2) by the high rate of cooling -- 30 to 50 degrees Centigrade an hour instead of 1.5 to 2.0 degrees Centigrade -- which allows the use of simple and less bulky equipment;

(3) by the use of filters instead of a centrifuge, which simplifies the process and makes it cheaper;

(4) by the high yield of oil -- 85 to 88 percent instead of 65 to 68 percent obtained from naphtha treatment;

(5) by the possibility of using this method to dewax almost any feed without preliminary refining.

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TABLE 54

RESULTS OF DEWAXING WITH MEK AND BENZOL

Characteristics of the products	Before dewaxing	After dewaxing	
		At -35° C	At -23° C
Specific gravity	0.885	0.898	0.891
Flash point, °C	223	232	--
Viscosity at 100° C	2.26	2.45	2.35
Solidification point, ° C	+7	-26	--20
Paraffin content, %	10.7	--	--
Index of viscosity	100	92	97
Yield of oil, %	--	80	86

126. Selection of Conditions for Dewaxing, and Conduct of the Process

The need for dewaxing any oil is determined by two conditions: (1) the characteristics of the stock and (2) the characteristics required of the finished oil.

The stock may be either residual or distillate, paraffinic or low-paraffin. Paraffin-free material, distillate and residual, does not require dewaxing, and its refining is limited to treatment with selecttive solvents. However, high-grade oil can be produced from wax stock only by dewaxing, which as a rule is performed after selective treatment of the stock. In case a high-resin stock is to be processed, it must under all circumstances be deasphalted first, with the order of the p rocesses in this case being: (1) deasphalting

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with propane, (2) dewaxing and (3) treatment with selective solvents.

A number of factors affect the dewaxing process: (1) the quality of the solvent, (2) the quantity of the solvent, (3) the dewaxing temperature and (4) the rate of cooling.

The characteristics of the solvents are the basic factor that affects all other dewaxing conditions.

(a) The solvent must completely dissolve oil and not dissolve paraffins and ceresins at the temperature selected for dewaxing.

(b) Ceresin and paraffin must separate out in the form of crystals which can be easily separated from the solution by filters or centrifuge.

(c) The solvent must not have corrosive properties, and must be nontoxic.

(d) The solvent must have a low freezing point.

One of the first and least successful solvents was naphtha. The disadvantages of this solvent have already been stated. The most widely used solvent is a mixture of MEK or aceton with benzol and toluol.

A mixture of dichlorethane with benzol, otherwise known as Bari-sol, is also widely used. This solvent has the same advantage as a mixture of MEK with benzol and toluol. A disadvantagous property of dichlorethane, however, is its higher toxicity.

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Liquid propane is often used for dewaxing. It is better than naphtha as a dewaxing agent, but is not as good as the other solvents we have considered. The principal disadvantage of liquid propane is the necessity for conducting the process under increased pressure (10-14 atmospheres). However, in some cases deasphalting and dewaxing with liquid propane is done simultaneously.

The world distribution of the various dewaxing processes on 1 January 1943 was a follows:

MEK (or acetone) with benzol and toluol	66%
Propane solvent	22%
Bari-sol solvent	7.5%
SO_2 + benzol	4.5%
TOTAL	100.0%

The quantity of solvent. Very small quantities of solvent do not allow the separation of the paraffins and ceresins from the solution. The greater the amount of solvents used, the more paraffins and ceresins are separated from the oil. However, an excessible amount of solvent is harmful since the ceresins and paraffins are soluable in it, even though only very slightly. A large amount of solvent adversely affects the quality of the oils, and in addition is disadvantageous for economic reasons.

Optimum rations are selected for each oil and for each solvent. The practical ratio of solvent to oil is 3:1 or 4:1.

The temperature of dewaxing depends on the solidification point of the finished oil and on the characteristics of the solvent. The temperature differential, as has already been noted, varies from 20 to 30 degrees for naphtha to 5 to 8 degrees for MEK.

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A high temperature differential characterizes the uneconomic nature of a process, since it requires large expenditures for cooling.

The conduct of dewaxing at very low temperatures makes it possible to produce low-soli difying oils, but at the same time reduces the index of viscosity. Thus, at a temperature of -35 degrees Centigrade an oil is obtained that solidifies at -26 degrees Centigrade, with an index of viscosity of 92, while at a temperature of -23 degrees Centigrade an oil solidifying at 20 degrees Centigrade is obtained with an index of viscosity of 97.

The rate of cooling also depends on the characteristics of the solvent and, depending on the latter, is selected within the range from 30 to 50 degrees Centigrade per hour.for MEK to 1.5 to 2.0 degrees Centigrade for naphtha. The rate of cooling affects the yield of finished oil and its characteristics.

The solidification point of the oil is controlled by the operation of the cooling and filtration unit (or of the centrifuge). The cooling of the oil solution is effected by the circulation of ammonia or of a brine coolant. The temperature of the cooling agent is regulated by the pressure in the ammonia system or by the quantity of the brine delivered. If the solidification point of the oils is higher than the norm (i.e., worse than the norm), the cooling temperature of the oils is reduced.

So as to diminish the loss of cold into the atmosphere, the insulation of the equipment must be maintained in best order. It is also necessary to watch out for any leakages through the stuffing boxes of the pumps and gate valves, and in the coils of

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the exchangers, so that the unprocessed oil does not enter the dewaxed oil, etc. It is also very important to watch out for the normal operation of the centrifuge and filters.

Before the process of dewaxing is performed, the so-called thermal treatment of the oil is carried out, i.e., the oil is heated to 60-70 degrees Centigrade so as to bring all the paraffins and ceresins into the liquid state. This is necessary so as to avoid the frequent separation of lumps containing certain amounts of oil when the oil is mixed as a solvent, thus leading to large oil losses.

The maximum yield of oil depends on the proper ratio between the solvent and the stock. A slight increase in the amount of the solvent introduced may result in increased yield of oil, but an excessive increase in the quantity of solvent leads to a higher solidification point of the oil, since part of the ceresin will then pass into the oil solution. This means that at the normal cooling temperature for the oil, in regulating the quantity of solvents (with the object of increasing the yield of oil) the solidification point of the finished oil must be checked. We have already said that a considerable amount of oil (15 to 25 percent) is carried over with the petrolatum. For this reason the petrolatum cake must be thouroughly washed on the filter in order to increase the yield of oil. In some refineries special units are constructed to deoil the petrolatum.

The minimum solvent losses depend on the normal operation of the entire regeneration system of the process unit, on the completeness of the stripping of the solvent from the oil (and also

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from the petrolatum), and on the completeness of the separation of the solvent from the condensate water. The complete order of the stuffing boxes and gaskets of the pump and gate valves must also be watched.

127. Deasphalting in Propane Solution

The treatment of high-resin tars and semi-tars by selective solvents does not produce good results, since it is not possible to produce oils with a good color and a low coke number in this way.

For this reason, before a high-resin stock is treated with selective solvents, it must first be freed of most of its asphaltic and resinous substances.

Flow chart (Figure 154). Liquid propane, preheated to 40 to 55 degrees Centigrade in the steam heat exchanger 2, is pumped through the mixer 3 together with the oil stock. The mixture of stock and liquid propane enters the inclined settling tank 4, where the mixture is settled by gravity and separated into two layers, an asphalt layer and an oil layer. The settling tank operates at a temperature of 45 degrees Centigrade and a pressure of 27 atmospheres.

The oil layer is heated in the steam heat exchanger 5A to 147 degrees Centigrade, and enters the oil column under a pressure of 13 atmospheres. The oil column consists of 3 superimposed columns: evaporating column 7, stripping column 7a and exhausting column 7b.

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The propane vapor is taken off overhead from the evaporating column and passed through condenser 8 into the propane storage tank 21. The oil, freed from the major part of the propane, enters the stripping column 7a, in which the pressure is lowered to 1.7 atmospheres. In the stripping column some further amount of propane is also driven off and passes through condenser 10, trap 13 (an apparatus for separating a gas from admixtures) and is then delivered by compressor 14 through cooler 8 into the propane storage tank. Finally, in the exhausting column 7b, the final removal of the last propane takes place.

The deasphalted oil is pumped into the storage tank from the bottom of the exhausting column. The asphalt layer from settling tank 4 is delivered to the asphalt column, which also consists of 3 columns: evaporating column 6, stripping column 6a and exhausting column 6b.

The asphalt layer is preheated to 157 degrees Centigrade in exchanger 5 and is delivered to the evaporating column, which operates under a pressure of 13 atmospheres at a temperature of 149 degrees Centigrade. The column receives the heat it requires for operation from steam still 19.

The propane is driven off through the top of the evaporating column, passes through cooler 8 and enters the propane storage tank. The asphalt layer flows into the stripping column, where an additional quantity of propane is stripped as a result of the reduction in the pressure to 1.7 atmospheres. The asphalt is then finally freed from propane in the exhausting column and is pumped into storage by pump 15. The path of the propane vapors

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driven off in the asphalt columns is the same as in the oil column.

Operating conditions: temperature and pressure. At a temperature of 38 to 60 degrees Centigrade, asphaltic and resinous substances are very slightly soluble in propane, while oil, paraffin and ceresins are completely soluble. As the temperature rises, the solubility of oil falls, and at 100 degrees Centigrade it is completely insoluble in propane. Deasphalting in practice is conducted at a temperature of 40 degrees Centigrade.

To keep the propane in the liquid state at this temperature in the asphalt settling tank, a pressure of up to 27 atmospheres is maintained.

TABLE 55

	Temperature, °C	Pressure, atmospheres
Asphalt settling tank	43	27
Outlet from oil preheater	147	--
Oil evaporating column	141	13.3
Oil stripping column	132	1.7
Outlet from asphalt preheater	157	--
Asphalt evaporating column	149	14
Asphalt stripping column	116	1.7

Ratio between propane and oil. With the addition of a small quantity of liquid propane to an oil stock, the latter becomes less viscous. The viscosity of the stock decreases as the quantity of propane is gradually increased and the moment arrives when the stock separates into a heavy high-viscosity asphalt and a mobile solution of oil in propane. The oil still contains a

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certain amount of asphaltic and resinous substances, while the asphalt still contains oil. When the quantity of propane is further increased, the oil layer is freed of the asphaltic and resinous substances, while the asphalt becomes more viscous and its melting point rises. Finally the moment arrives when the further addition of propane does not extract any more asphaltic and resinous substances from the oil. The proportion of propane to oil used in practice is on the order of 7:1 to 10;1.

Good results are also obtained at the refineries by using the propane-propylene fraction instead of propane.

The results of deasphalting a concentrate of Koschagyl' crude with propane and the propane-propylene fraction, in comparison to contact treatment, are presented below.

TABLE 56

	Stock	Treatment with propane (1:4 at 25° C)	Treatment with propane-propylene (1:6 at 40° C)	Treatment with acid (15%) and earth (20%)
Specific gravity	0.942	0.916	0.919	0.923
Viscosity, E_{100}	15.4	6.33	7.17	8.67
Coke, %	5.79	2.3	2.68	3.27
Brenken flash point, °C	296	306	308	309
Yield of products, %	100	68.5	72	51

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The yield of oil on deasphalting with propane or the propane-propylene fraction is considerably higher than with acid-clay treatment, and its quality is better. On treatment with propane, not only asphalted and resinous substances can be removed from the oil but also the paraffin and ceresins (if the process temperature is lowered to 38 degrees Centigrade). Concurrently, the solidification point of the oil is lowered to 15 to 18 degrees Centigrade.

128. Compounding and Mixing of Oils. The Use of Additives

Even the most perfect treatment does not always produce oils of the required characteristics. The requirements for oils have increased so greatly that it has become necessary to use certain other methods for producing high-grade oils. Moreover, the deep refining of distillates or residues to produce oils of the required characteristics is accompanied by high losses. This is why refiners have started using special substances called additives which upon addition to oils in small quantities considerably improve the characteristics of oils.

At the present time a large number of additives are known to improve various qualitative indices of oil: some of them reduce the solidification point, others raise the index of viscosity, others increase the stability, still others increase the lubricant properties of oils, etc.

The best known of the additives that reduce the solidification point of oils are paraflow, santopur and voltol. For instance, the addition of 1 percent paraflow lowers the solidification point of the oils produced from Bibi-Eybat crudes 25 degrees to 30 degrees Centigrade. Santopur gives the same result

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when used in the amount of 0.1 to 0.5 percent. Voltol must be added in considerably large amounts (2 to 4 percent).

Exanol and voltol are used to raise the index of viscosity. Three percent exanol raises the index of viscosity of a naphthene base oil from 26 to 99, and 5 percent raises it to 111; 6 percent of voltol increases the index of viscosity from 65 to 106. To improve the lubricant qualities of oils, small amounts of animal or vegetable fats are added.

Mixing and compounding oils. By mixing two kinds of oils of different viscosity an oil of a third kind can be prepared. For instance, Diesel oils are obtained on mixing bright stock with SU machine oil; MC winter aviation oil is obtained by mixing MK summer aviation oil with spindle oil, etc. Oils are mixed in oil-boilers by heating slightly and circulating with a pump.

Compounding, or mixing mineral oils with vegetable oils, is distinguished from ordinary mixing. For instance, marine oils are prepared by mixing heavy machine oil with 10 to 20 percent rape oil.

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CHAPTER THIRTEEN

THE AUXILIARY SERVICES IN AN OIL REFINERY

Each oil refinery, besides the main process units that refine crude, also has a number of shops and services for serving the process units. They include: (1) the tank system, (2) pumping stations, (3) filling and discharging racks, (4) steam-power system, (5) electric power (6) water supply, (7) drainage system and traps, (8) machine-repair shops, (9) the laboratory and (10) fire protection service.

The shops and units are arranged in the plant for convenience in serving the basic process units and to meet the rules of fire prevention.

129. The Tank System

The tank system of a refinery consists of several separate farms; a crude-oil tank farm, distillate farms (a separate one for each process unit, a finished production farm (also separate for each process unit) and a commercial tank farm.

The crude tank farm consists of large tanks with a capacity ranging from 4,000 to 10,000 cubic meters, and is usually located 1 to 3 killemeters and more from the refinery.

The intermediate crude-oil tank sections consist of 2 or 3 tanks of 500 to 1,000 cubic meters each, which are located alongside the process units. The intermediate tanks allow the gravity settling of water and sediment and the correct accounting of the quantity of processed crude.

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The distillate sections and the finished product sections consist of small gauging tanks, each of 100 to 200 cubic meters capacity. The capacity of the gauging tanks is calculated to cover the requirements of a single shift for each product. At the end of each shift the product or distillate is changed over to another gauging tank, while the product produced by the last shift is settled, gauged and analyzed in the laboratory. If the characteristics of the product agree with the assigned specifications, it is pumped into the commercial tank. If it does not, it must be rerun.

The commercial-tank farm consists of large tanks, each with a capacity of over 1,000 cubic meters. The finished product is shipped out of the plant from each of these tank farms.

129. Discharging and Filling Docks

As a rule, each refinery has its own railroad spur with a rack, i.e., an arrangement in the form of a group of standpipes for filling finished products into tank cars and discharging crude from tank cars into the refinery crude-oil storage tanks.

Every refinery does not have an unloading railroad rack. Crude is delivered by pipe lines to refineries located in oil-producing regions. Refineries located on rivers receive their crude by water transport (barges). A so-called crude-oil machine is installed at the bank of the river. This is a barge with powerful centrifugal pumps, by which the crude is pumped out of the barges into the oil-storage tanks of the refinery. Such refineries do not need a railroad unloading rack.

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There are two methods of discharging crude at loading racks: overhead and bottom. Heavy crudes and mazut are discharged from the bottom of the tank carsinto channels located between the rails. The crude flows by its own gravity into tanks set in the earth beside the trestle, and is then pumped into the crude-oil storage tank. Light crudes are discharged through the upper hatch of the tank car by means of standpipes. In the standpipes and pipe lines a vacuum is created by a pump, the crude is drawn into the pipe line and then flows out of it by gravity into the storage tank.

Every refinery must have a loading rack. There are several dozen standpipes on the rack, which continue the corresponding product lines, and are provided with flexible hose connections the ends of which are introduced into the tank cars. Each product has its own battery of standpipes. They are arranged in such a way that a train, consisting of 20-30 tank cars, may be simultaneously filled with any product.

131. The Pumping System.

The piping system and pumps play a very great role in the work of a refinery, since they are the principle and almost the only method for moving crude and the products obtained from it. Each tank farm has its own pumping station and each product and distillate has at least two pumps, an operating pump and an emergency pump.

Besides the pumping stations connected with the corresponding tank farms, there are a number of pumping stations at each refinery which handle all the internal pumping to a process unit, and all the pumping out of distillates and finished products into the distillate

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receptacles and finished-product tanks.

A petroleum distillation unit consists of the following pump houses: the cold-oil pumping station, where the crude-oil pumps are located; a hot-oil pumping station where the reflux pumps are located; a product pumping station where the pumps for handling the distillates and finished production, etc, are located.

132. Drainage System and Traps

When the tanks are cleaned, part of the crude oil and products is carried away by water and sediments; in the operation of the mixing condensers and dehydrators, part of the products may also be carried away with water, and small leaks are possible in the operation of the condensers, coolers, pumps, pipe systems, etc. There are outlets leading to the drainage system for collecting the water with the oil products carried along, in every tank farm, at every production unit, on the racks and from the pumping stations.

The drainage system consists of a wide ceramic or concrete pipe with dimensions calculated for handling all waste water from the condensers and coolers, the steam condensate and all possible wastes. The drainage pipe is laid sloping towards the disposal-point from which the waste enters the general drainage system only after proper purification (acid wastes, for instance, must under all circumstances first be neutralized).

An oil trap for recovering lost oil products is standard equipment in every refinery. A trap is constructed in a form of a rectangular tank, and has a number of baffles that do not go all the

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way to the bottom. The wastes entering the trap from the drainage system move very slowly in it, with a velocity no higher than 0.5 meter/minutes; during this motion the liquid becomes separated into two layers: water at the bottom, oil product at the top. Only the water passes under the baffle, while the oil product remains in the trap in the product section, from which it is pumped into a tank.

133. The Power System

Steam Supply

Steam is used at refineries in large quantities, from 0.5 to 2 tons for each ton of crude throughput. It is used for motive power to run the pumps, for process purposes in the rectifying columns, furnaces and stills, as a fire extinguishing medium, and for heating the shop buildings. If the steam supply is interrupted, the work of the whole refinery is brought to a standstill. For this reason every refinery has its own steam system.

The boiler plant of a refinery always has a reserve capacity of 25-30 percent. For instance, a refinery has a daily throughput of 1,000 tons of crude, with an average consumption of 0.7 ton of steam per ton of crude, i.e., 700 tons of steam a day. If boilers with a daily capacity of 50 tons are selected, 14 such boilers must be installed, together with 4 reserve boilers, or 18 boilers in all. Consequently, 4 boilers may be under repair and cleaning or held in reserve.

The steam boilers at oil refineries are of various types, but usually their capacity is not great since if accident should put

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more boilers out of commission at the same time than the number assigned to the reserve, the supply of steam would be considerably reduced, which would affect the normal operation of the refinery. The steam pressure at different refineries ranges from 4 to 10 atmospheres, sometimes ever running up to 25 atmospheres. Steam is superheated at the process units themselves, in superheaters installed in the convection chambers of the furnaces.

Electricity Supply

Some of the pumps are driven by electric motors (centrifugal and electric-drive piston pumps). Almost all the control instruments and automatic control devices are electrical. The fans, recyclers, mixers and machines are driven by electric motors. The refineries receive the necessary electric energy from outside sources, but they also have emergency Diesel electric power plants (in case of accident to the city power station).

Water Supply

The consumption of water at oil refineries is very great and may reach 40-50 tons per ton of fully processed crude (straight-run distillation, production of lube oils and cracking).

Different qualities of water are required for different purposes. For instance, steam boilers need pure water with only a small amount of salts, but water less pure than this may be used for coolers and condensers. For this reason every refinery has a triple system of water supply: (1) pure water for steam boilers, treatment units and sanitary purposes; (2) sea or river water for coolers, condensers, etc, and (3) water under high pressure for the

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system of fire mains and standpipes.

The fire mains must be completely independent of the other two systems.

Special pumping-stations are set up at the shore of the river or sea to supply refineries with water. From there, powerful pumps pump water through 2 or 3 ten or twelve inch lines to the refinery. Where sea or river water is unavailable, deep wells are drilled from which water is pumped to the refineries.

134. Fire Safety at Refineries

An oil refinery is one of the most dangerous undertakings with respect to fire hazard, since it must deal with highly inflammable oil products and gases.

To prevent the outbreak of fire at a refinery, or to liquidate it if it should occur, the following rules must be observed.

(a) In the design of a plant, the process units must be arranged precisely in accordance with the norms of the fire safety rules as must the equipment in the several process units; according to these norms the equipment is located in a definite order, with a definite spacing between the units.

(b) Each tank or group of tanks must be surrounded by an earth embankment of such dimensions that in case of overflow of the product from the tank all of it will remain inside the embankment.

(c) The tanks and equipment at the process units must be provided with means of extinguishing fires (steam standpipes with steam

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hoses), foam extinguishers and water-extinguishing facilities (fire hydrants).

(d) A large number of water hydrants must be provided throughout the whole refinery.

(e) The drainage system must be provided with hydraulic traps, that is, wells with baffles that do not go all the way to the bottom; a water seal in the drainage system is thus created by these baffles, and a fire arising anywhere in the drainage system will be localized by such seals.

(f) All refinery workers must be familiar with the fire protection instructions and know how to use the simple fire-fighting facilities: fire extinguishers, sand, felting, water and steam hoses.

(g) The process units must be supplied with the necessary number of sand boxes, fire pails, fire shovels, fire extinguishers and felting.

Every refinery has its own fire company, which must always be fully ready for duty.

If fire breaks out at a refinery, various methods of extinguishing it are used, depending on size and location of the fire.

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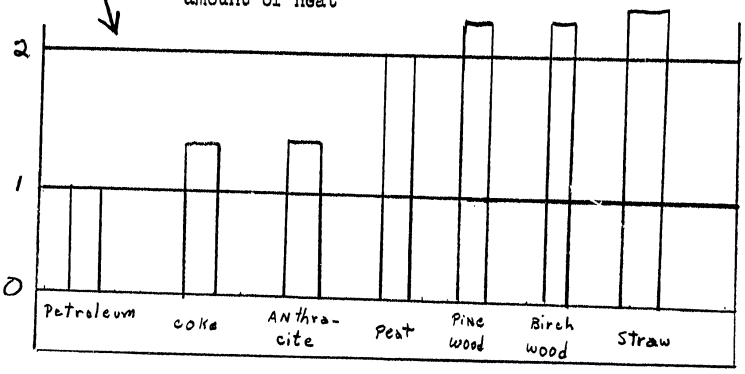
APPENDIX

[p 4] Figure 1. The still of the Dubinin brothers

1. heater 2. cooler 3. receiver

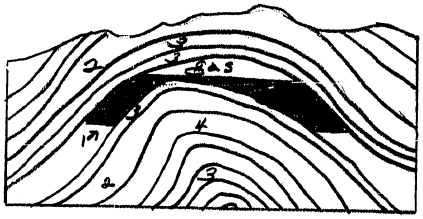
Figure 2. A modern oil refinery. [Photo]

[p 5] Figure 3. Comparison of the consumption (in units of weight) of various fuels required to develop the same amount of heat



[p 7] Figure 4. Distribution of gas, petroleum and water in the folds of the earth's crust

1. oil 2. sand 3. clay 4. mixture



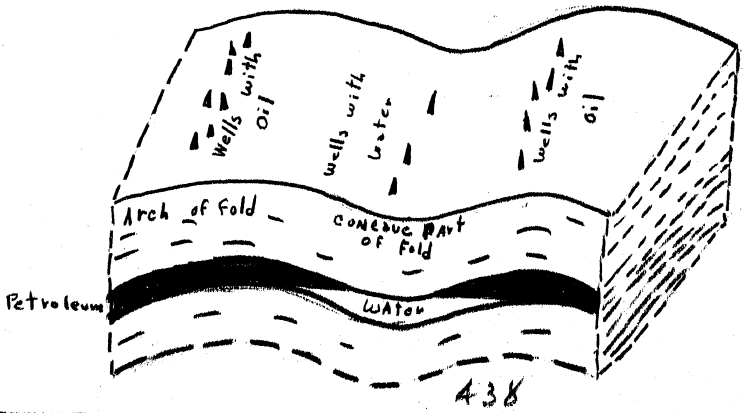


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Figure 5. Aerometer

[p 12] Figure 6. Specific-gravity balance

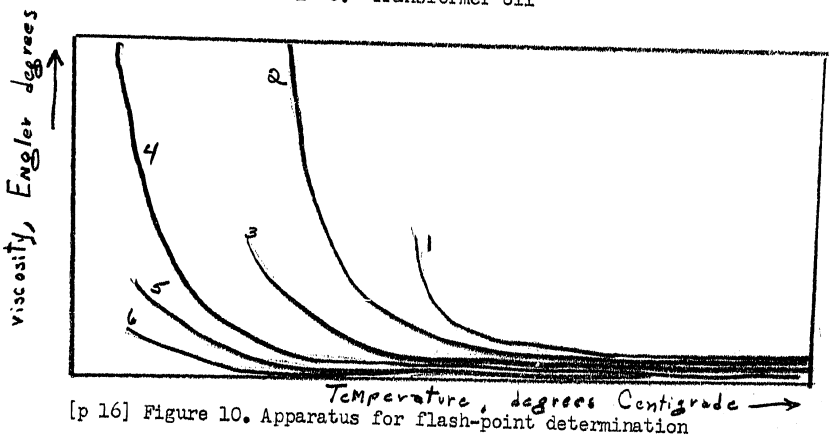
Figure 7. Pycnometers

[p 13] Figure 8. Viscosimeter. [Photo]

1. Internal reservoir for product being tested 2. External reservoir (bath) 3. Agitator handle 4,5. Thermometers 6. Wooden plunger 7. Measuring flask 8. Ring burner

[p 14] Figure 9. Curves of variation of viscosity with temperature

1,2. Residual cylinder oils 3. Vapor 4. Lubricant-oil mazut 5. Rape (Vegetable) oil 6. Transformer oil



[p 16] Figure 10. Apparatus for flash-point determination

[p 18] Figure 11. Apparatus for freezing-point determination

[p 19] Figure 12. Colorimeters for determining the color of petroleum products [Photo]

[p 22] Figure 13. Apparatus for determining the fractional composition of light oils

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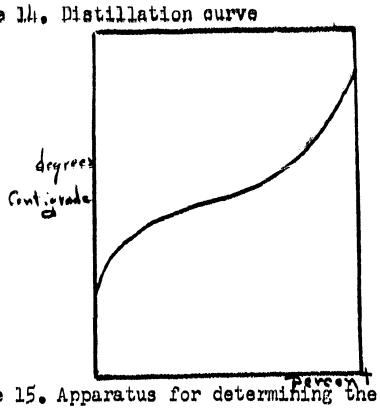
[p 23] Figure 14. Distillation curve



[p 30] Figure 15. Apparatus for determining the water content of petroleum products

1. 500-milliliter flask 2. Receiver-trap
3. Reflux condenser 4. Outlet tube

[p 31] Figure 16. The four-stroke cycle

[p 32] Figure 17. Effect of the compression ratio on engine power and fuel consumption

A. Cylinder volume

B. Volume of compressed mixture

A : B Compression ratio

at compression ratio	5:1	6:1	7:1
the developed HP of engine is	114	125	135
the fuel consumption in kilograms per HP hour is	0.24	0.21	0.20

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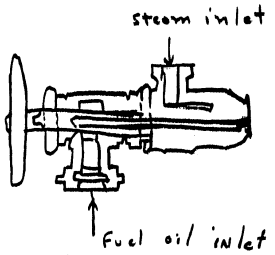
[p 37] Figure 18. Apparatus for vapor-pressure determination [Photo]

[p 41] Figure 19. The Shukhov burner



[p 42] Figure 20. Apparatus for coke determination [Photo]

[p 46] Figure 21. Pumps

Top: Diagram of piston pump Center: Cross-section of piston pump Bottom: Cross-section of centrifugal pump

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[p 48] Figure 22. Pipe in pipe heat exchanger [Photo]

[p 49] Figure 23. Single-pass heat exchangers

Figure 24. Multi-pass sectional shell and tube heat exchanger (a) longitudinal section (b) transverse section

[p 52] Figure 25. General view of a coil-in-box cooler

1. welded iron tank 2. coil, of cast iron tubes 3. fittings 4. header 5. standpipes delivering water 6. water overflow pan 7. stairs and platform 8. hatch for cleaning

[p 53] Figure 26. Mixture condenser with dehydrator

1. vapor inlet 2. vapor inlet 3. gas discharge 4. distillate discharge

Figure 27. External view of selective coil-in-box coolers [Photo]

[p 54] Figure 28. Spray coolers [Photo]

[p 55] Figure 29. Core of heat exchanger [Photo]

[p 57] Figure 30. Radiant-and-convection type pipe still with vertical flow of gases

[p 61] Figure 31. Heat balance of a pipestill

1. enters with fuel 2. lost to surrounding medium in lower half of firebox 3. transferred by radiation from the combustion mirror and from the side walls 4. lost to surrounding medium in upper half of firebox. 5. transferred by radiation from the gas stream 6. transferred by convection to the radiant tubes 7. lost to surrounding medium in convection chamber 8. transferred to upper convection section 9. used for superheating steam 10. given up to the two lower convection

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shafts 11. passes off in the smokestack
12. used for preheating combustion air, returning with it into the firebox

[p 62] Figure 32. Types of pipe stills
1. Pile pipe still 2. Pipe still with removable firebox 3. Convection pipe still 4. Convection pipe still 5. Radiant-and-convection pipe still 6. Cross-over pipe still 7. Modern radiant-and-convection pipe still with vertical flow of gases 8. same 9. Soviet pipe still with bank of floor tubes 10. Radiant and convection pipe still with horizontal flow of gases 11. Two-chamber pipe still

[p 63] Figure 33. Pipe still bracing

[p 64] Figure 34. Flat arch of pipe still [Photo]

[p 65] Figure 35. Return bend

[p 70] Figure 36. Laboratory still with Gadaskin fractionating column:
1. still 2. column with aluminum packing
3. dephlegmator 4. thermometer 5. cooler
6. receiver

[p 71] Figure 37. Rings for packed columns [Photo]

Figure 38. Construction of bubble plates of a rectifying column

[p 72] Figure 39. Diagram showing fractionation of a three-component mixture

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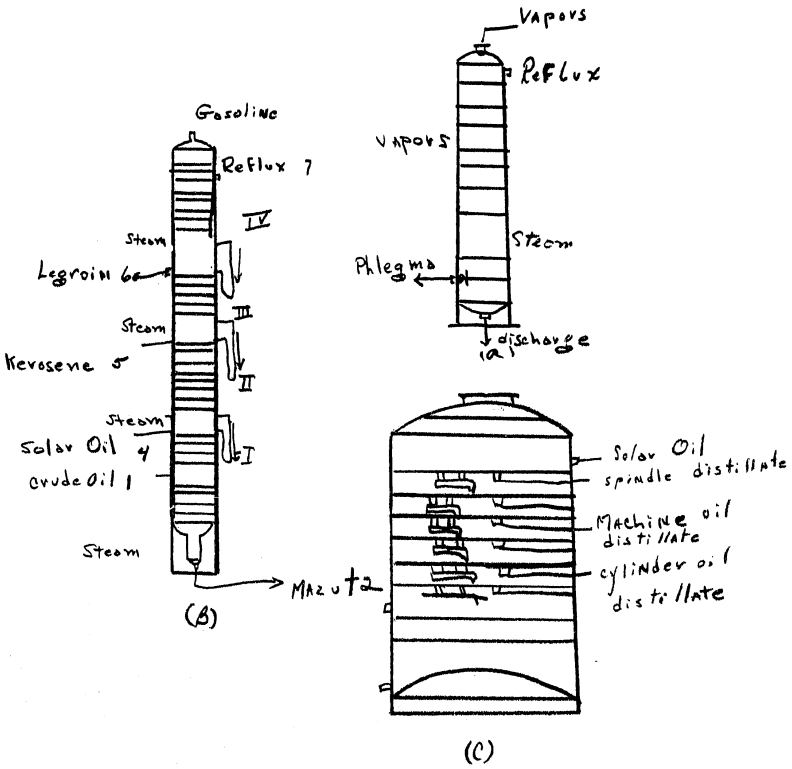
[p 73] Figure 40. Rectifying columns

(a) packed column (b) atmospheric column

(c) vacuum column



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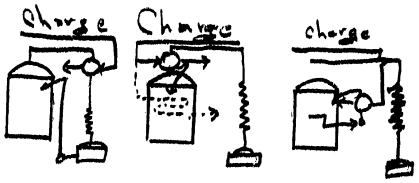
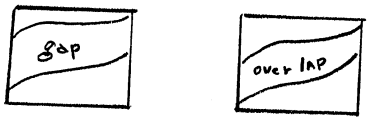
[p 74] Figure 41. Methods of refluxing

1. Cold reflux 2. Hot reflux 3. Circulating reflux



[p 76] Figure 42. Distillation curves of two adjacent distillates



[p 78] Figure 43. Tray with rectangular bubble caps [Photo]

[p 79] Figure 44. Round bubble cap

Figure 45. Tray with grooved bubble caps

A. structure of tray B. longitudinal section of the bubble tower C. flow diagram of the bubble caps

[p 80] Figure 46. Trays with hexagonal bubble caps [Photo]

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[p 80] Figure 47. General scheme of the structure of a barometric condenser:

A. mixture condenser B. barometric tube C. well (overflow) with hydraulic seal

1. vapor inlet 2. water inlet 3. racks 4. gas discharge

Figure 48. Vacuum ejector

1. nozzle 2. steam inlet 3. vapor and gas inlet 4. discharge of vapor mixture

[p 82] Figure 49. Control panel of a pipe-still unit [Photo]

[p 83] Figure 50. Thermocouples

a. thermoelectric circuit

1. copper wires 2. cold couple 3. hot couple 4. galvanometer

b. thermocouple with porcelain insulation

c. thermocouple with flanged protection tube

Figure 51. Recording pyrometer [Photo]

[p 84] Figure 52. Manometer

[p 85] Figure 53. Diagram of float-type level indicator for steel tank

[p 86] Figure 54. Liquid-level controller

A. pipe through which liquid is pumped out of tower a. liquid level b. discharge cock 1. globular float (floats in float chamber) 2. chain connecting levers 3 and 6 3. lower lever 4,5. valves for cleaning out chamber 6. upper lever with counterpoise on end 7. valve

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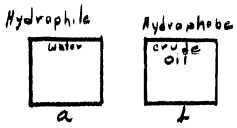
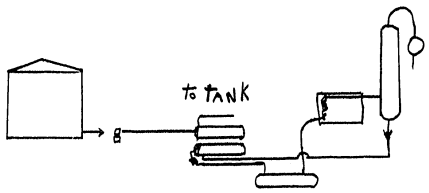
[p 89] Figure 55. Types of emulsions

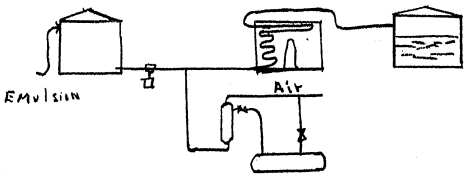
a. Hydrophile emulsion b. Hydrophobe emulsion



[p 90] Figure 56. Diagram of a crude distillation plant using a dehydrator-sediment trap (center of diagram) to tank



[p 91] Figure 57. Diagram of demulsification unit



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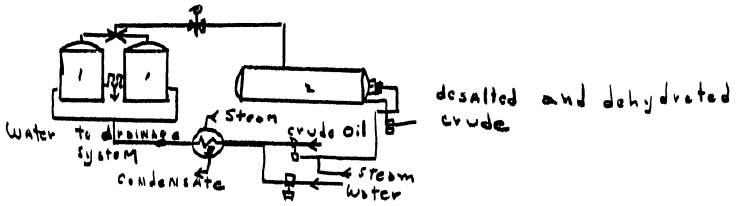
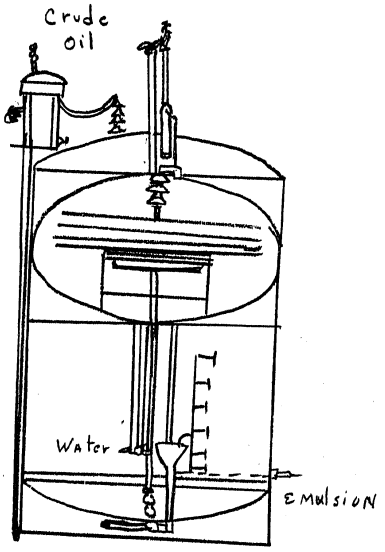
[p 91] Figure 58. Diagram of electrical dehydration unit

1. electrical dehydrator 2. settling tank

3. preheater



[p 92] Figure 59. Electrical dehydrator (section)



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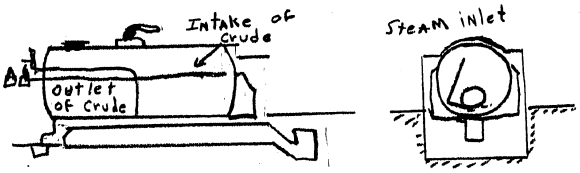
[p 93] Figure 60. General view of old type of shell still battery: [Photo]
1. Kerosene stills 2. Overhead pipes 3. Dephlegmators 4. Lube oil stills

[p 95] Figure 61. Shell-still plant for the distillation of crude:
1. preheater 2. shell still 3. dephlegmator 4. condenser 5. cooler 6. scrubber 7. cut house a. pipe to condenser b. crude supply to still c. hot mazut supply to preheater

Figure 62. Shell still



[p 97] Figure 63. General view of shell-still battery with rectifying column [Photo]

[p100] Figure 64. Lube-oil shell-still (longitudinal section
1. Firebox 2. Protective brick arch 3. Path of flue gas from flue I to side flue II and then to lower flue III 4. Still domes 5. Line to vacuum equipment

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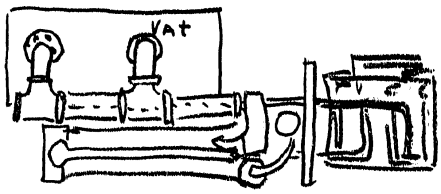
[p 101] Figure 65. System of vapor condensation and distillate cooling in a lube-oil still

1. still domes 2. air cooling tubes (the path of the vapors is indicated by arrows) 3. dephlegmator 4. exhauster 5. vacuum line 6. water cooled coolers



[p 101] Figure 66. Water-jet vacuum injector of an oil battery (exhauster:

1. entrance of the vapors into dephlegmator
2. water inlet 3. balancing line

[p 106] Figure 67. General view of a single-flash unit [Photo]

[p 107] Figure 68. Single-flash atmospheric unit with radiant-and-convection pipe still with horizontal flow of gases:

1. pipestill 2. rectifying column 3. stripping column 4. gas-oil cooler 5. spray-type heat-exchangers 6. ligroin coolers 7. mazut coolers 8. kerosene coolers 9. mixture condenser 10. water separator 11. dehydrator 12. crude-oil pump 13. mazut pump 14. reflux pump

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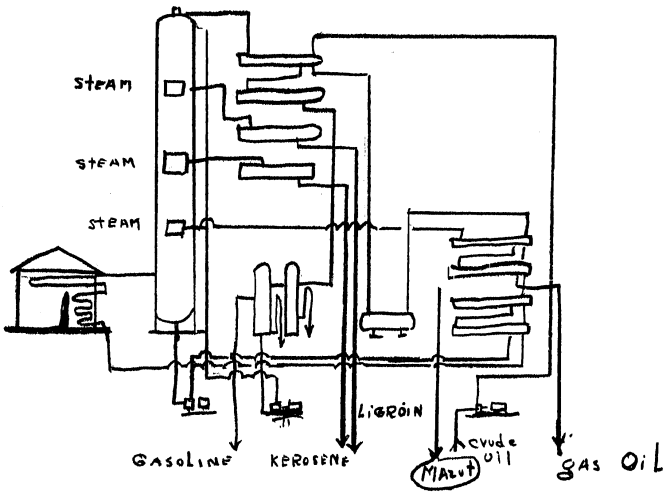
[p 108] Figure 69. General view of single-flash unit [Photo]

[p 109] Figure 70. Atmospheric single-flash distillation unit:
1. distillate heat-exchangers 2. sediment settler 3. heat exchanger 4. pipe still 5. fractionating column 6. cooler 7. water separator



[p 111] Figure 71. Transverse section of Soviet pipe still (atmospheric).

[p 111] Figure 72. Longitudinal section of Soviet pipe still (atmospheric)
1. explosion window

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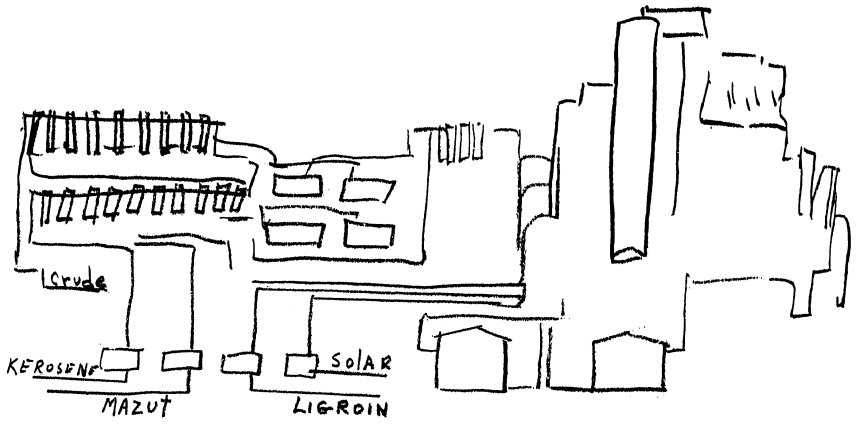
[p 112] Figure 73. The Soviet pipe still plant

1. circulating reflux heat exchangers 2. kerosene heat exchangers 3. mazut heat exchangers 4. dehydrators 5. pre-evaporator 6. rectifying column 7. stripping column 8. gasoline condenser 9. scrubber-dehydrators 10. pipe stills 11. vapor heat exchangers



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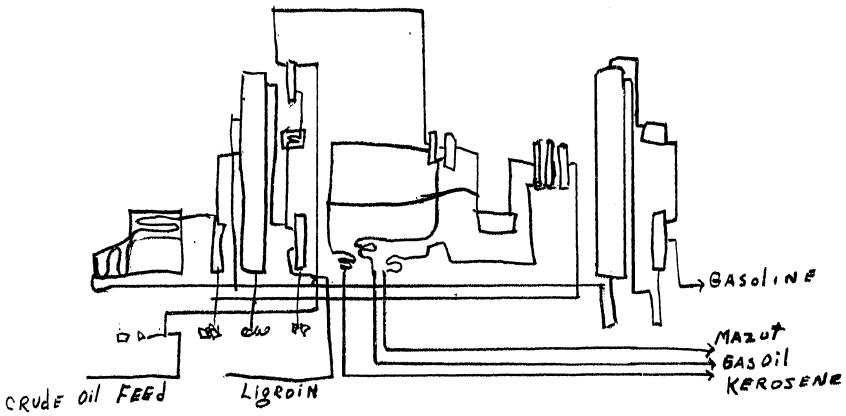
[p 114] Figure 74. Two-flash atmospheric unit

1. vapor heat exchangers 2. heat exchangers 3. sediment settler 4. first column 5. condenser 6. dehydrator 7. pipe still 8. second column 9. stripping column 10. cooler



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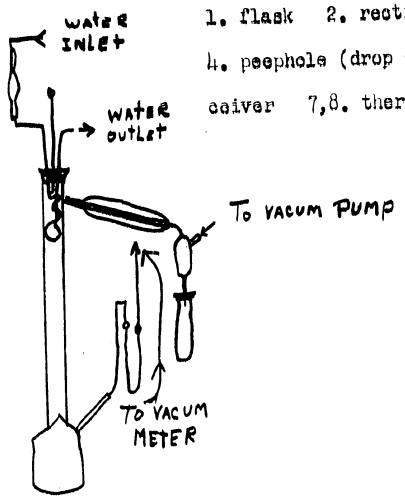
[p 121] Figure 75. Still for ITK-method distillation:

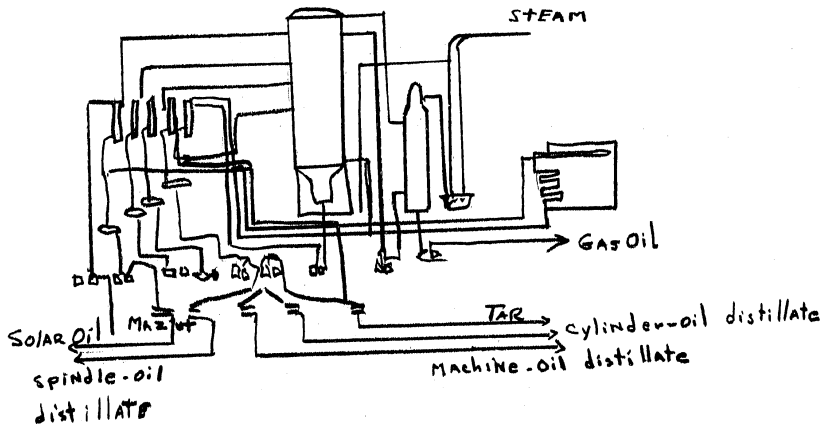


1. flask 2. rectifying column 3. dephlegmator 4. peephole (drop counter) 5. cooler 6. receiver 7,8. thermometers 9. manometer

[p 137] Figure 76. General view of vacuum units [Photo]

Figure 77. A vacuum unit with radiant-and-convection pipe-still with vertical flow of gases

1. heat exchangers 2. pipe still 3. vacuum column 4. barometric condenser 5. steam jet 6. vacuum receivers 7. coolers



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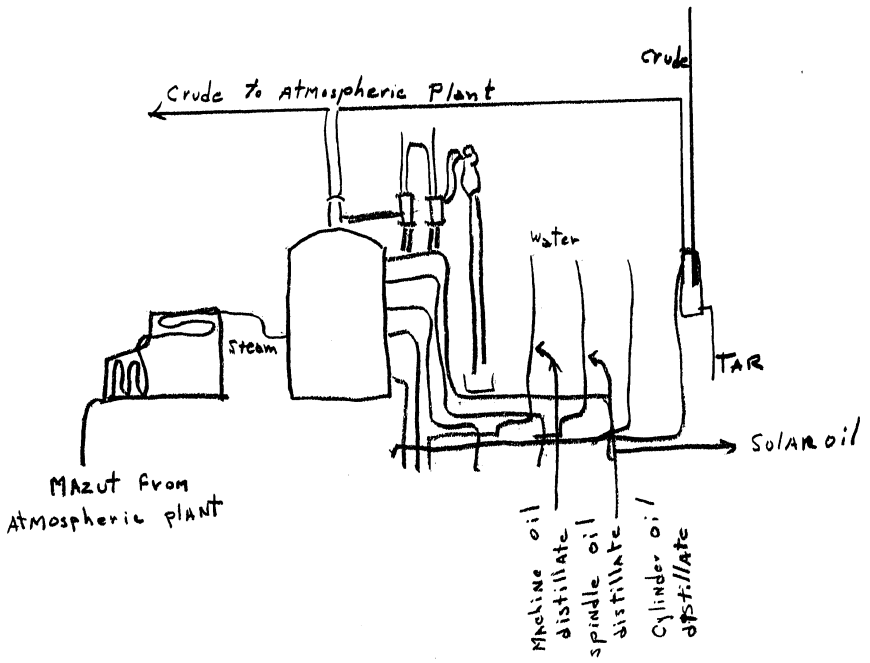
[p 139] Figure 78. Vacuum unit with radiant-and-convection pipe still with horizontal flow of gases: 1. pipe still 2. vacuum column 3. partial condenser 4. solar oil condenser 5. coolers 6. tar heat-exchanger 7. steam jet



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[p 140] Figure 79. The Soviet Vacuum Unit:

1. charging pump 2,3 vapor heat exchangers 4. tar heat exchangers 5. pipe still 6. column 7. hot-oil pump 8. cooler 9. barometric condenser 10. ejector 11. well 12. hot-air fan 13. air preheater 14. flue

_____ Distillate

_____ Mazut

_____ Tar

---------- Water

[p 141] Figure 80. General view of Soviet vacuum unit [Photo]

[p 151] Figure 81. Scheme of simplest cracking plant

[p 152] Figure 82. General view of cracking plant [Photo]

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[p 152] Figure 83. Flow sheet of a cracking plant with horizontal reaction chambers

1. rectifying column 2. reaction chamber 3. evaporator 4. cooler and condenser for cracked distillate 5. mixing reservoir 6. hot-oil pump feeding the cracking coil 7. pump to feed charge to plant and to pump it through the column overhead coil acting as dephlegmator 8. charge tank 9. return flow of product into reservoir after cooling at top of column 10. tubular coolers 11. gas trap 12. outlet of finished cracked distillate 13. outlet of cracked mazut 14. pipe still heater

[p 156] Figure 84. Scheme of low-pressure coil of Winkler-Koch cracking plant:

1. outlet of feed 2. inlet of feed 3. steam into coil 4. drop-out line 5. discharge 6. steam

[p 156] Figure 85. Scheme of high-pressure cracking coil:

1. evaporator 2. drop-out line 3. from low-pressure cracking coil 4. reducing valve 5. inlet to coil 6. purging steam 7. outlet from coil 8. testcock 9. discharge 10. purging steam 11. emergency steam

Figure 86. Evaporator (left) and rectifying column (right) of a Winkler-Koch cracking plant

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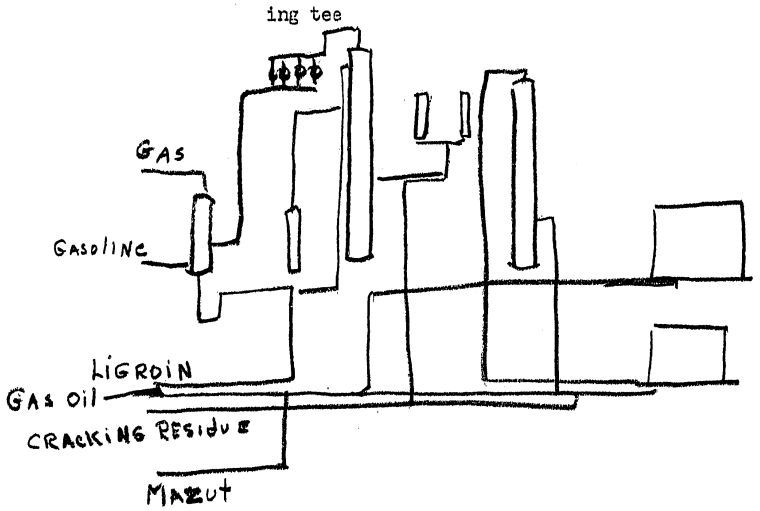
[p 157] Figure 87. Flow sheet of a Winkler-Koch cracking plant

1. charging pump 2. vapor heat-exchangers 3. low-pressure cracking coil 4. high-pressure cracking coil 5. evaporator 6. rectifying column 7. condenser 8. gas trap 9. ligroin cooler 10. gas-oil cooler 11. cracking-residue cooler 12. hot-oil pump 13. stripping column 14. five-outlet pump for gas-oil reflux 15. mixing tee



[p 159] Figure 88. General view of high-pressure cracking coil [Photo]

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[p 161] Figure 89. The photograph shows a model of the duplex two-coil system cracking plant [Photo]

At the back of the scene are four single pipe stills, of which the two at the outside are designed for mild cracking, and the two in the middle for deep cracking. Each pipe still has a smokestack, at the base of which are the regenerator for pre-heating combustion air and the fans for delivering it to the pipe still, drawing the flue gas out of the pipe still, and recirculating it. In front of the pipe stills is the fractionating equipment: the evaporator (near the center), the first column and second column. The gasoline vapor and gas is conducted by an uninsulated head pipe from over-head of the second column of each pipe still into a coil-in-box type condenser located on the roof of the neighboring building. This structure is the hot-oil pumping station that feeds the pipe still. The final cooling of the gasoline and gas takes place in horizontal tubular coolers (one for each unit) which may be seen on the photograph, in front of the hot-oil pump buildings. Here the gas separator is also located (the extreme column) where the gasoline and gas are separated, and the absorber (the high column near the center) into which the gas from the gas separator is directed.

The cracked residue passes from the evaporators into the additional evaporators which may be seen

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at the center of the photograph beyond the control building. The latter is marked T on the plan. The area around which the additional evaporators are grouped is reserved for the hot-oil pumps pumping out the cracked residue. On the roof of this part of the building there are coil-in-box condensers for the solar-oil fractions vaporized in the additional evaporators. Coil-in-box coolers in front of the control building serve to cool the cracking residue.

All the control of the process is handled from the control room. Here are located the pumps, the leads to the reducing valves and the indicators of the control instruments. The control instruments themselves are located for reasons of fire protection outside the wall of the control room in a long, low annex which is visible in front of the control room.

On the right-hand side of the model there is a separate plant for stabilizing the cracked gasoline from the duplex two-coil cracking plant. Here are the pumping station, control room, gas separator and stabilization column.

In contrast to most of our two-coil cracking plants, this model shows a plant not equipped with treatment towers.

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[p 162] Figure 90. Framework of the evaporator and columns of the two-coil cracking plant

[p 163] Figure 91. Additional evaporator (vertical section)

1. connecting pipe for feeding the cracked residue into the evaporator 2. baffle plates 3. bubble-cap plates 4. overhead pipe 5. connecting pipe for feeding cracking stock 6. connecting pipe for discharging cracking stock 7. connecting pipe for supplying superheated steam 8. connecting pipe for discharge of the evaporated and heavier residue 9. connecting pipe for discharging the vapors of the light fractions 10. manhole

[p 164] Figure 92. Flow sheet of Nefteproyekt two-coil cracking system

1. mild cracking furnace 2. deep cracking furnace 3. evaporator 4. rectifying column No 1 5. rectifying column No 2 6. pressure-distillate condenser 7. final coolers 8. first gas trap 9. absorber 10. tank belong to absorber 11. additional evaporator 12. condensers 13. dehydrator 14. accumulator 15. heat exchangers 16. transit hot-oil pump 17. hot-oil pump made by Sumsk plant 18. cooler for cracking residue 19. phlegma from the stabilizer 20. pressure distillate to vapor-phase treatment.

[Diagram on next page]

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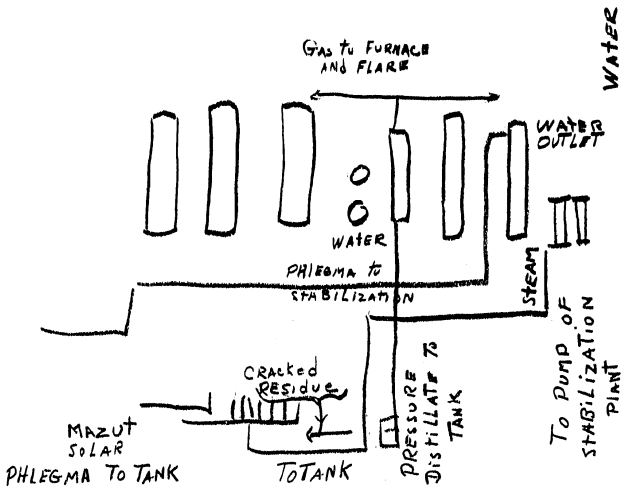
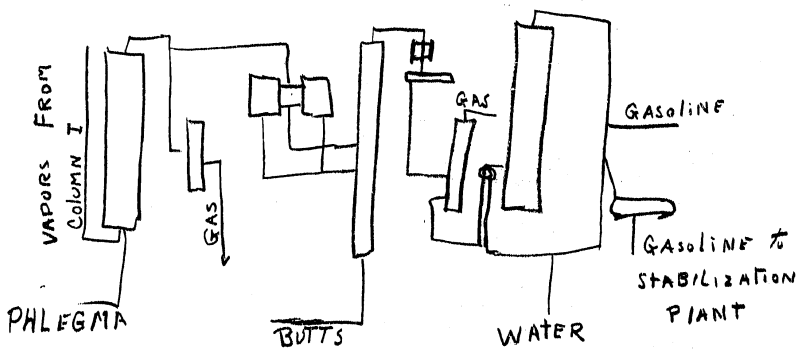
[p 164] Figure 92.



[p 165] Figure 93. Vapor-phase treatment with clays

1. rectifying column 2. treatment towers 3. additional column 4. condenser for treated gasoline 5. final cooler 6. second gas trap 7. butts cooler 8. mixers 9. alkali-wash column 10. tank of stabilization plant 11. pressure-distillate condenser 12. first gas trap



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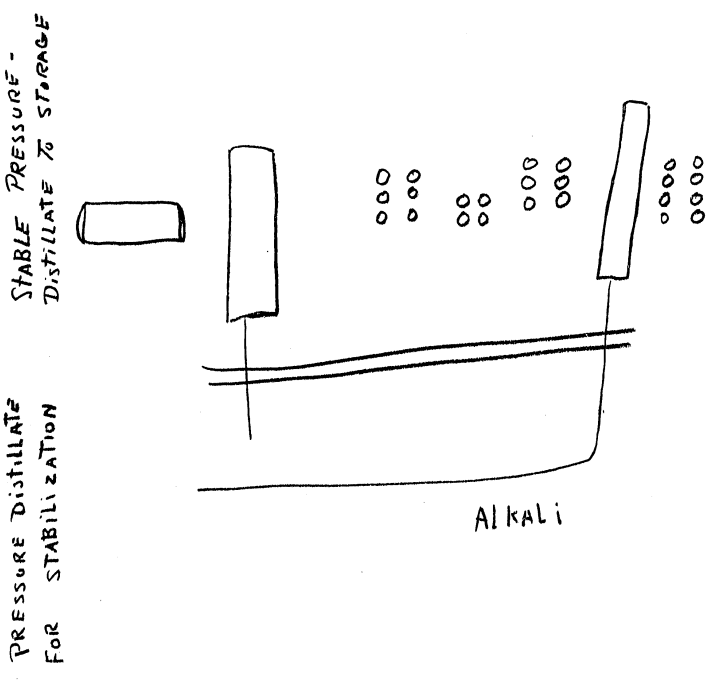
[p 167] Figure 94. Flow sheet of Nefteproyekt-system stabilization plant

1. stabilizer 2. alkali column 3. feed tank (unstable pressure distillate) 4. reflux tank 5. coolers 6. stripping stills 7. condenser-cooler 8. heat exchangers 9. charging pumps 10. alkali pumps 11. reflux pumps 12. liquid gas to pressure tanks 13. phlegma from second column 14. phlegma to heat exchangers 15. mixer for pressure distillate and alkali 16. gas to furnaces



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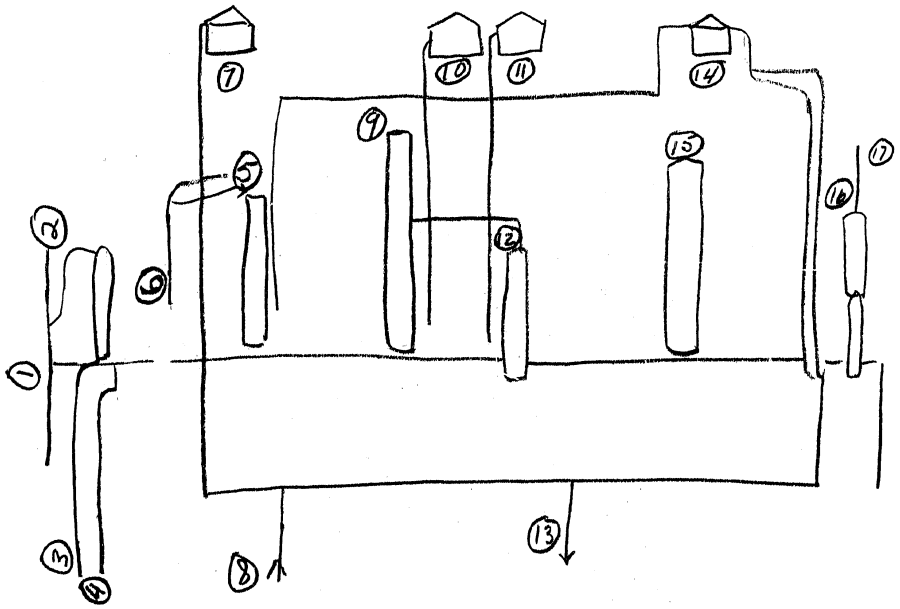
[p 169] Figure 95. Flow sheet of combined cracking plant

1. gas to fuel 2. straight-run gasoline stabilizer 3. straight-run gasoline 4. cracked gasoline 5. straight-run column 6. gas and H_2S 7. reforming furnace 8. feed 9. low-pressure evaporator and column 10. visbreaking furnace 11. gas-oil cracking furnace 12. residue evaporator 13. residue 14. recyling furnace 15. high-pressure evaporator and column 16. dry gas 17. stabilizer and absorber



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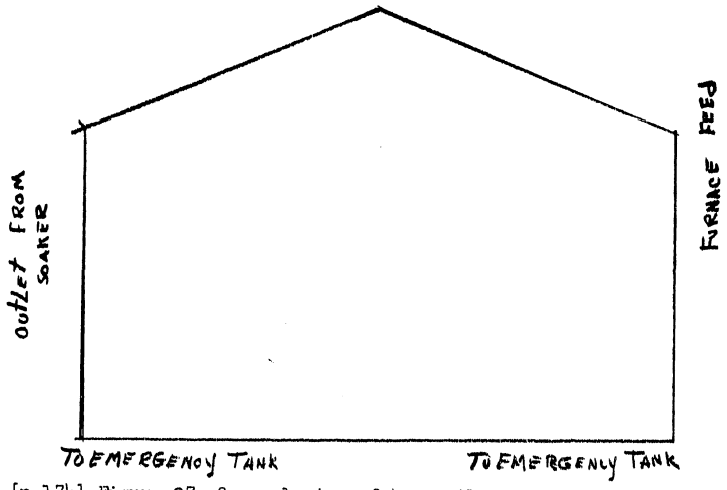
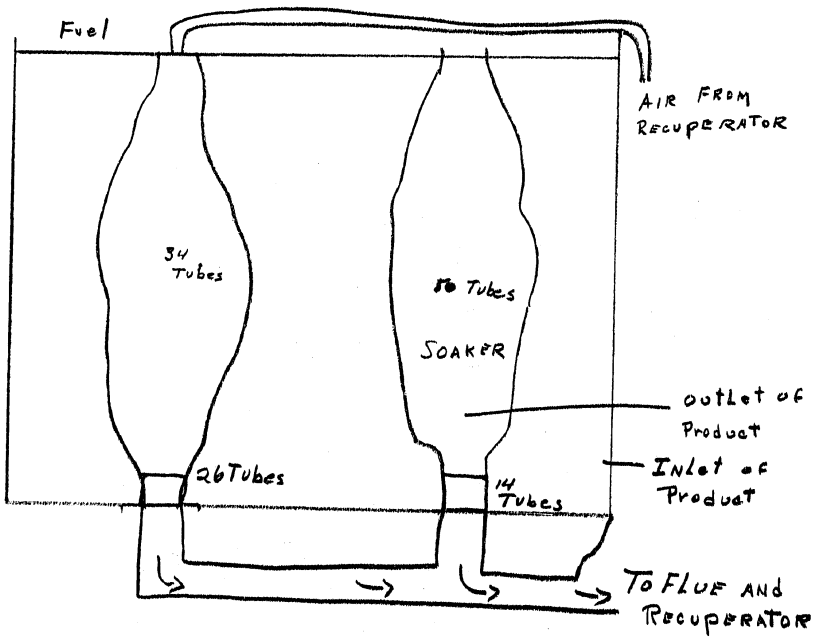
[p 171] Figure 96. Mild-cracking furnace



[p 174] Figure 97. General view of two-coil cracking plant with vertical reaction chamber [Photo]

[p 175] Figure 98. Section of two-chamber cracking furnace:

1. special high-grade refractory 2. I class refractory



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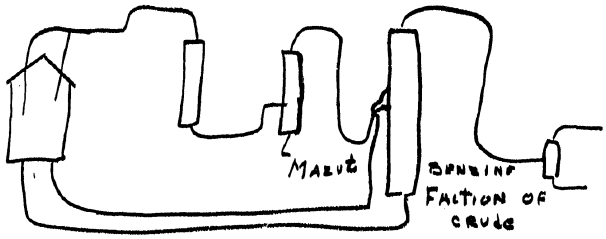
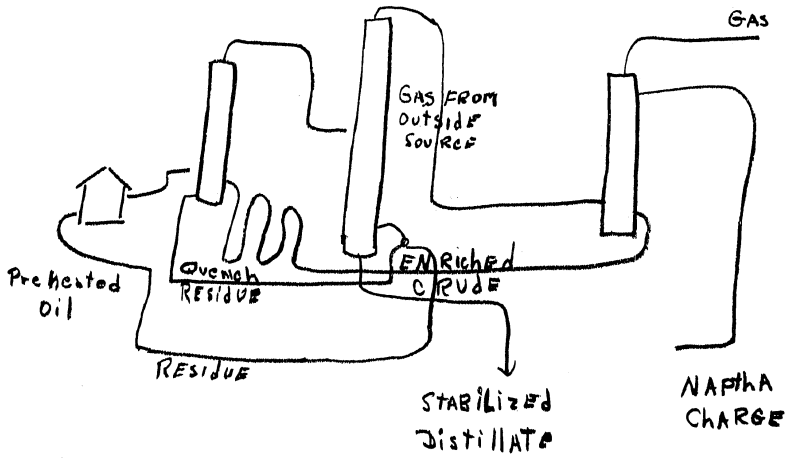
[p 175] Figure 99. Two-coil selective cracking unit;

1. mild-cracking furnace 2. gas-oil cracking furnace 3. reaction chamber 4. additional evaporator 5. rectifying column



[p 178] Figure 100. Scheme of gas-reversion cracking

1. furnace 2. combined separator and rectifying column 3. overhead reflux 4. gas-oil reflux 5. dehydrator 6. stabilizer 7. absorber



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[p 187] Figure 101. Pneumatic drill [Photo]

Figure 102. (a) outside view of adjustable hammer head

1. joint 2. hammer head

(b) outside view of cutter with miniature turbine

3. miniature turbine 4. milling cutter

[p 188] Figure 103. Cleaning the tubes of a high pressure cracking coil with the miniature turbine [Photo]

[p 191] Figure 104. Furnace with cast-iron retorts and condensation system:

1. feed inlet 2. furnace 3. hydraulic 4. air coolers 5. water coolers 6. exhauster 7. scrubbers 8. inlet of scrubbing oil 9. discharge of scrubbing oil 10. gas to gasholder

[p 192] Figure 105. Furnace with fireclay retorts

A. retorts B. burners C. hydraulic

[p 193] Figure 106. Generator

1. fuel supply 2. feed inlet 3. air inlet

4. flue 5. outlet to hydraulic 6. hydraulic

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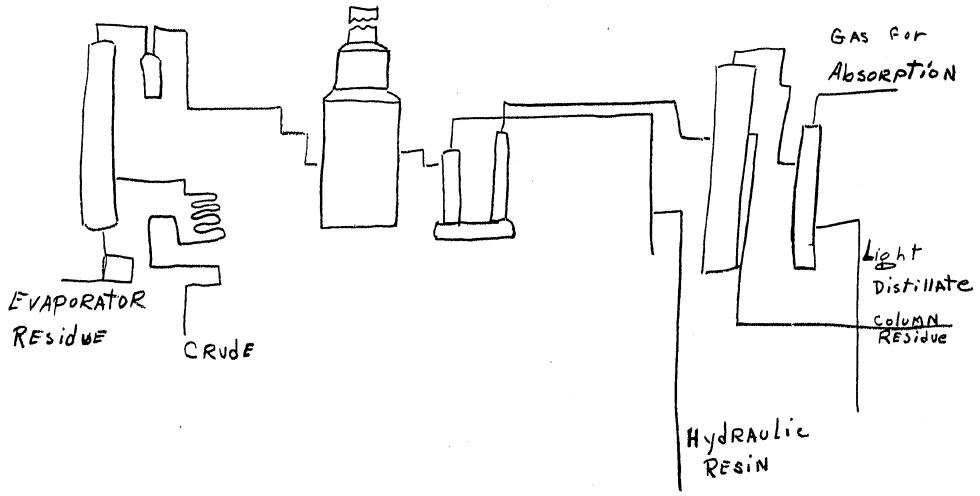
[p 195] Figure 107. Schematic flow sheet of pyrogenous pipe-still plant

1. heat exchanger 2. pipe still 3. evaporator 4. separator 5. reaction chamber 6. cooler for evaporator residues 7. hydraulic tank 8. sludge pan 9. storage tank 10. cooler of hydraulic resin 11. rectification column 12. condenser 13. gas separator 14. cooler for column residues



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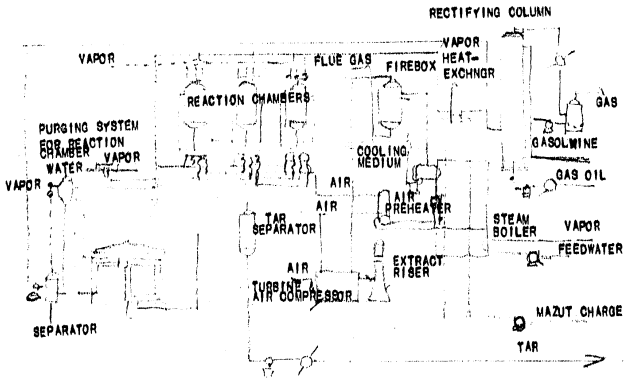
[p 196] Figure 108. Diagram of a Unit for Catalytic Aromatization

1. crude -- heavy ligroin 2. still 3. gas for regeneration of the catalyst 4. spent gas after regeneration 5. compressor 6. catalytic chamber 7. recycling gas 8. gas separator 9. stabilization column 10. dry gas 11. benzene in storage tank 12. redistillation column 13. residue outlet

[p 202] Figure 109. Schematic flow sheet of static-catalyst catalytic cracking



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[p 203] Figure 110. Sketch of reaction chamber with static catalyst

Diagram: (top) Inlet of heat carrier

(middle) outlet of vapors and flue gas to vacuum

(bottom of tank) outlet

(bottom) Perforated plate for catalyst

Inlet of vapors and gas

Discharge pipe for heat carrier

[p 204] Figure 111. Section of catalytic reaction chamber (left, vertical; right, horizontal)

[p 205] Figure 112. Chart of operation of three pairs of catalytic cases of a static-catalyst plant

1. cracking 2. change-over and steam purging before regeneration 3. regeneration 4. change-over and steam purging after regeneration

Diagram: (vertical) cases nos 1 and 2, 3 and 4, 5 and 6

(horizontal) Time in minutes

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[p 211] Figure 113. General view of catalytic cracking plant with powdered catalyst [Photo]

[p 212] Figure 114. Catalytic cracking with powdered catalyst

1. heat exchanger 2. feed preheater 3. injector for feed 4. distributing header 5. reactor 6. cyclone separator of reactor 7. catalyst feed pipe 8. valve on spent-catalyst line 9. reaction product to column 10. air compressor 11. spent catalyst and air to regenerator 12. regenerator 13. distributor 14. cyclone separator of regenerator 15. stripping column for catalyst regeneration 16. regenerated catalyst 17. spent catalyst 18. cooler for catalyst 19. fractionating column 20. pump for residue 21. steam boiler-utilizer 22. apparatus with electrodes

Diagram: (extreme left) Air (left of 21) air water (left of 18) water air

(right side - top to bottom) gasoline and gas catalytic gas oil feed

(center; above 8) steam for purging

(bottom) fresh catalyst residue from column

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[p 214] Figure 115. Scheme of clay-burning furnace; stripping column for regenerator of catalyst

1. fusion furnace for metal or salts 2. pump with reservoir 3. separator 4. charging bin for clay 5. regeneration chamber 6. clay discharge 7. fan

Figure 116. Details of inner construction of clay-burning furnace [Photo]

[p 215] Figure 117. The catalytic cracking process with moving catalyst

1. heat exchangers 2. pipe still 3. evaporator 4. tar cooler 5. reactor 6. evaporator for spent and regenerated catalyst 7. regenerator 8. rectifying column 9. stripping column 10. gas tray 11. compressor

Diagram: (left) tar (right of 7) flue gass (above 10) gas

(bottom, left to right) air water gas oil fuel feed

ligroin
gasoline

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[p 216] Figure 118. Schematic diagram of section of catalytic cracking and catalyst regeneration

1. regenerated catalyst 2. bucket conveyor for regenerated catalyst 3. cyclone separator 4. level indicator 5. catalyst in hopper 6. discharge of catalyst fines 7. level indicator 8. flue gas 9. regenerator 10. catalyst duct to reactor 11. spent catalyst 12. bucket conveyor for spent catalyst 13. differential level controller 14. regenerated catalyst 15. flue gas 16. steam 17. separator 18. vapor 19. air 20. vapor and air 21. reactor 22. cracking products 23. feed 24. steam 25. spent catalyst 26. vapor settler 27. catalyst from hopper 28. regenerated catalyst 29. fire-box-air-preheater 30. fuel 31. air blast 32. air 33. water in drum 34. drum of steam generator

[p 218] Figure 119. Two-stage plant with moving catalyst [Photo]

[p 221] Figure 120. Absorption plant to extract gasoline from gas [Photo]

[p 222] Figure 121. Flow sheet of stabilization of raw natural gasoline

1. charging pump 2. heat exchanger to preheat charge to feed temperature 3. stabilized-gasoline cooler to cool it before discharge into commercial storage tank 4. condenser for reflux vapors 5. reflux collector 6. stripping still 7. level controller 8. thermoregulator 9. back-pressure controller 10. flow regulator 11. stabilizer

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[p 223] Figure 122. Flow sheet of plant at Ishimbay

1. absorber 2. scrubber 3. heat exchanger 4. preheater 5. demethanizer 6. stripping column 7. lean absorbent tank 8. column condensers 9. separator 10. storage tank for crude benzene 11. tank for fractionation feed 12. preheater for depropanizer feed 13. depropanizer 14. tank for stabilizer feed 15. stabilizer 16. cooler for stabilized gasoline 17. tank for butane-column feed 18. butane column 19. cooler for n-butane

Diagram: (under 7) to recompression (right of 5 and 6) Steam (right of 14) stabilized natural gasoline (left of 19) n-butane (right of 19) isobutane

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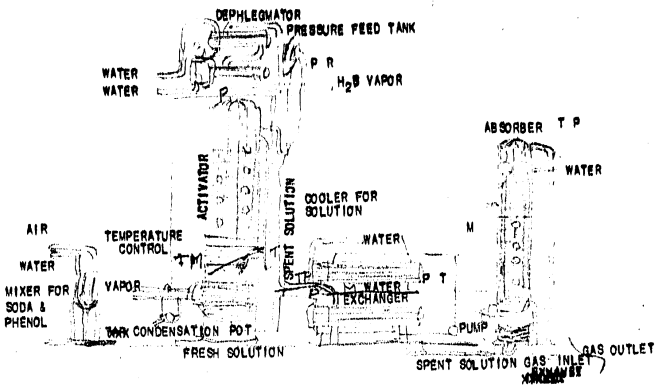
[p 226] Figure 123. Scheme of phenol scrubber

T thermometer R recording thermometer

P manometer M recording liquid consumption meter



[p 229] Figure 124. General view of polymerization plant [Photo]

[p 230] Figure 125. Catalytic polymerization plant

1. charging compressor 2. furnace 3. graduated water tanks 4. pump 5. reactors 6. coolers 7. column 8. stripping still 9. condenser

Diagram: (left top) gas to fuel (left center) gas to recycle (bottom middle) gas from cracking plant (over 8) steam (under 6) gasoline to storage

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[p 233] Figure 126. Sulfuric-acid Alkylation Plant

1. cooler 2. reactor 3. acid settler
4. alkali settler 5. preheater

Diagram: (under 4) alkali (14) steam (under 3) circulating acid
waste acid discharge
(right side, top to bottom) coolant fresh acid feed propane
alkylate alkylate residue normal butane
(under 15) isobutane

[p 234] Figure 127. Types of Contactors for Alkylation Plants

1. Tank-type contactor 2. hatch 3. injection nozzle 4. mechanical agitator 5. steam turbine motor

Diagram: (Left half —) (top) Feed of circulating mixture
(bottom) Drainage (left) outlet of circulating mixture (right)
hatch inlet.
(Right half) (left top) inlet of acid (left bottom) Feed of hydrocarbon mixture (in body) circulation (right, top to bottom)
outlet of collant liquid outlet of mixture of acid and hydrocarbons Pump discharge

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[p 238] Figure 128. Hydrofluoric acid alkylation

1. alkylation stock 2. bauxite column for drying stock 3. reaction chamber 4. circulating isobutane 5. recycled acid 6. acid settler 7. acid 8. acid treatment system 9. hydrofluoric acid 10. recovery of spent acid 11. water 12. petroleum product to be dissolved in acid 13. propane 14. depropanizing column 15. deisobutanizing column 16. debutanizing column 17. alkylate 18. aviation alkylate 19. alkylate rectifying column 20. heavy alkylate

[p 242] Figure 129. Catalytic Alkylation of Benzol with Propylene

1. heat exchanger 2. fired preheater 3. reactors 4. depropanizer 5. benzol column 6. rerun column 7. accumulators 8. fired preheaters

Diagram: (bottom left) propane-propylene fraction (bottom right) benzol (center under 7) propane (right under 7) alkylate (center bottom) benzol (bottom right) polymers

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[p 244] Figure 130. Details of tank sweating chamber (in each tank a separate detail is shown for clarity)

1. window 2. support for tanks 3. grating with screen supporting it 4. feed pipe from charging supply pipe 5. paraffin discharge pipe 6. water coils and pipe delivering water 7. water coils with water discharge pipe 8. location of pipes for filling box during time of sweating 9. steam coil for heating chambers

[p 247] Figure 131. Mixing tanks for treatment of petroleum products [Photo]

[p 251] Figure 132. Continuous acid-alkali treatment of gasoline and ligroin

1. feed tank 2-4. acid settlers 5-7. alkali settlers 8. treated-product tank 9. air lift for spent acid 10. air lift for fresh acid 11. fresh-acid tank 12. alkali-solution tank 13-17. injectors

Diagram* (left) acid settlers (right) alkali settlers

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[p 252] Figure 133. Tube-type injector mixer

Diagram: (left) distillate inlet (top and bottom) reagent inlet

[p 253] Figure 134. Plant for sulfuric-acid treatment of cracked distillates [Photo]

[p 254] Figure 135. Mixer

1. entrance of petroleum products 2. reagent inlet 3. outlet of mixture 4. primary mixing chamber 5. screw propeller 6. shearing baffle plates 7. shell of contactor 8. metal gasket 9. circulation tube 10. straightening baffles 11. atomizing (diffusion) baffles 12. removable valve, removed for cleaning contactor 13. pipe for draining contents and sampling 14. extra feed inlet for product to be treated 15. gasket 16. spring 17. sleeve 18. shaft 19. stuffing box 20. ring 21. discharge opening 22. coupling 23. coupling bolt 24. bracket supporting motor 25. electric motor

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[p 254] Figure 136. Centrifuge

1. mixture of gasoline and acid 2. inspection port 3. collector of treated distillate 4. acid sludge collector 5. outlet of treated distillate 6. outlet of acid sludge 7. rotor 8. motor

[p 255] Figure 137. Schematic diagram of three-stage sulfuric-acid treatment

1. centrifuge 2. separator 3. mixer 4. heat exchanger 5. filters 6. flowmeter 7. air lift 8. regulator 9. pump 10. acid-sludge pump

Diagram: (top) first stage second stage third stage (4) water (under 5) acid sludge for treatment (then down) for neutralization (bottom) fresh acid

[p 258] Figure 138. Schematic flow sheet of atmospheric and vacuum redistillation with steam heating

1. treated gasoline 2. heat exchanger 3. stripping still 4. atmospheric column 5. stripping still 6. tank 7. condenser-cooler 8. receiver 9. vacuum column 10. stripping still 11. condenser 12. receiver-dehydrator 13. to steam jet 14. to gasoline storage tank 15. outlet to polymer tank 16. outlet to commercial storage tank 17. vapor

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[p 260] Figure 139. Schematic diagram and equipment for acid-alkali treatment of kerosene

1. acid mixer 2. alkali mixer 3. tank for alkali liquor 4. tar tank 5. pump 6. acid egg for acid 7. tar discharge pipe 8. outlet from acid mixer into alkali mixer

Diagram: (top) (left of 1) air H_2SO_4 (right of 1) water

distillate

(left of 4) air water vapor (right of 4) organic part, to fuel

(right of 2) air NaOH water

(bottom) (left) distillate (top and right) air

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[p 261] Figure 140. Continuous treatment of kerosene

1. measuring tank for acid 2. measuring tank for alkali 3-5, 13-15. mixers 6-8, 16. acid mixers 17. settling columns 10-12, 18. alkali settlers

Diagram: (top left) alkali settlers (top right) acid settlers (bottom) water (right bottom) kerosene

[p 263] Figure 141. Equipment for acid-alkali treatment of lubricating oils

1. acid mixer 2. alkali mixers 3. wash vat 4. drying tank 5. acid egg

[p 272] Figure 142. Outside view of treatment tanks [Photo]

Figure 143. Tanks for clay treatment

[p 273] Figure 144. Acid-contact lube-oil treatment plant

1. acid mixer 2. acid oil settler 3. large mixer 4. clay silo 5. furnace of distillation unit 6. evaporator 7. column 8-9. coolers 10. small mixer 11. filter press 12. gauge tank for intermediate oil 13. press 14. pump

[See diagram on following page]

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Diagram: (right of 1) air acid (bottom) oil acid sludge
(center) clay

[p 274] Figure 145. Filter press [Photo]

[p 275] Figure 146. Lubricating-oil clay-filtration unit

1. transporter 2. filter 3. overflow tank

[p 277] Figure 147. Schematic-flow diagram of furfurol-treatment plant for lubricant oil

1. feed stock enters plant 2. pump delivering furfurol 3. steam preheater for distillate 4. steam preheater for furfurol 5. extraction tower 6. receiver for raffinate solution 7. receiver for extract solution 8. pump delivering raffinate solution for distillation 9. tube heater for raffinate solution 10. main vacuum column for distilling raffinate solution 11. pump delivering raffinate to stripping column 12. steam stripper for raffinate 13. heat exchanger using heat of raffinate to heat furfurol 14. pump delivering extract solution for distillation 15. tube heater for extract solution 16. atmospheric column for distillation of extract

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solution 17. heat exchanger 18. vacuum column for distillation of extract solution 19. mixture of furfurol vapors 20. furfurol condenser 21. vacuum receiver for dry solvent 22. pump delivering dry solvent to tank 23. steam stripper for extract 24. mixture of wet-solvent vapors to condenser 25. circulation of extract solution through heater and back to vacuum column 26. pump for circulation of extract and delivering it to stripper 27. condenser for wet solvent 28. receiver for wet solvent 29. pump delivering dry solvent to tank 30. dry vacuum pump 31. cooler 32. receiver for wet solvent 33. solvent cooler 34. tank for wet solvent 35. pump delivering finished extract to tank 36. pump delivering wet solvent to fractionator 37. furfurol drier 38. furfurol fractionator 39. settler for solvent 40. reflux condenser 41. tank for pure solvent 42. pump delivering finished raffinate to storage tank

[p 280] Figure 148. Schematic flow sheet of double-solvent treatment E-1 and E-2. extractors E-3. collector N-1. evaporator for extractor N-2. additional evaporator N-3. column for solvent N-4. vacuum column N-5. collector for solvent P-1. evaporator for raffinate P-2. additional evaporator P-3. column for solvent P-4. vacuum column P-5. collector for propane W-1. water

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evaporator W-2. evaporator

1-9. section of extractors 10. mixer 11. heat exchanger 12. condenser 13. compressor 14, 15. preheaters 16. heat exchanger 17. furnace 18. condenser 19. cooler 20. receiver 21. coolers 22. heat exchanger 23. preheaters 24. cooler

Diagram: Propage (over n-1, over N-2, over p-2 at left, over P-1, in 2 tanks over 12, and under w*1) Reboiler (under N-1, under P-1)

Selecto (over N-3, over P-3, and under N-5) Vapor (left of 21, and under P-4, vertical and right bottom of diagram) Extract (right of 21) raffinate (underP-4) reflux condenser (over P-2 at right) cooler (over 1) extract solution (over N-5) raffinate solution (right of E-3) Gaging tank for propane (under P-5) vacuum cooling system (under 10) feed stock (in tank at lower right corner of diagram).

[p 284] Figure 149. Flow diagram of countercurrent extraction

1. solvent 2. distillate 3. raffinate 4. extract 5. drops of distillate

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[p 287] Figure 150. Flow sheet of dewaxing by propane in naphtha solution

1. orifice mixer 2. preheater 3. reservoir 4. cooling towers 5. pressure feed tank 6. centrifuge 7. inlet of ammonia 8. cooling plant 9. receivers 10. cooler 11. pump 12. to collecting reservoir 13. stripping plant for petrolatum

Diagram: (under 1) solvent feed (under 12) diluted feed (under 11) petrolatum

(12) to reservoir (over 13) petrolatum

[p 288] Figure 151. Centrifuge [Photo]

[p 289] Figure 152. Scheme of centrifuge operation

A. position of mixture at rest B. same, in operation

[p 290] Figure 153. Flow sheet of plant for dewaxing with MEK-benzol-toluol mixture.

1. Arrival of stock for mixing with solvent 2. steam preheater for solution of stock 3. water cooler for solution of stock 4. heat exchanger for cooling 5. coolers cooling by

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direct expansion of ammonia 6. feed tank for filters 7. filter (vacuum) 8. filtrate receiver 9. screw device to remove cheese paraffin 10. heater for cheese paraffin 11. settler to remove water from cheese paraffin 12. flue-gas cooler 13. vacuum pump 14. gasholder 15. admission of inert gas 16. cooler for solvent 17. cold filtrate to heat exchanger 18. filtrate to evaporator 19. evaporator for filtrate 20. evaporator for petrolatum 21, 22. water coolers 23. solvent tank 24. solvent for washing filtrate 25. solvent for dissolving stock 26. filtrate to stripping column 27. stripping column for filtrate 28. dewaxed oil 29. petrolatum for distillation 30. petrolatum to stripping column 31. stripping column for petrolatum 32. cheese paraffin to storage 33. piping system gathering steam and solvent vapor 34. water cooler 35. separator (to separate water and solvent) 36. solvent to storage 37, 38. watery ketone to fractionator 39. fractionator 40. discharge of water into drainage system 41. supply of water 42. spent steam to heater 43. live steam to heater 44. high-pressure steam

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[p 295] Figure 154. Flow sheet of plant for removing tar by propane

1. feed 2. steam heat-exchanger 3. mixer 4. settler 5. heat exchanger 6. evaporator column for asphalt solution 6a. exhausting section of column 6b. stripping section of column 7. lubricant oil evaporating column 7a. exhausting section of column 7b. stripping section of column 8. cooler-condensers 9,10. condensers 11. feed cooler 12. dehydrator 13. trap 14. compressor 15. pump to draw out asphalt 16. pump to draw out oil 17. pump to supply propane 18. charging pump 19, 20. steam stills 21. propane tank

Diagram: (left of 2) vapor (over 4) oil solution (under 4) asphalt solution (under 1) tar feed stock (right of 15) asphalt (left of 17) oil (right of 21) delivery of propane

[p 299] Figure 155. Steel tanks

a tank with conical roof b tank with spherical roof

[p 303] Figure 156. Earthen levee around tanks

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